PB84-229723

Application of an Analysis Protocol to Identify Organic Compounds Not Identified by Spectrum Matching. Part 2: Appendices

Research Triangle Inst.
Research Triangle Park, NC

Prepared for

Environmental Research Lab., Athens, GA

Aug 84

U.S. Department of Commerce
National Technical Information Service
NTIS

PB84-229723

EPA-600/4-84-072b
August 1984

APPLICATION OF AN ANALYSIS PROTOCOL
TO IDENTIFY ORGANIC COMPOUNDS
NOT IDENTIFIED BY SPECTRUM MATCHING
Part 2: Appendices

by

Joan T. Bursey
Research Triangle Institute
Research Triangle Park, North Carolina 27709

Contract No. 68-03-2867

Project Officer

Walter M. Shackelford
Analytical Chemistry Branch
Environmental Research Laboratory
Athens, Georgia 30613

ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
ATHENS, GEORGIA 30613

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

1. REPORT NO.
EPA-600/4-84-072b

2.

3. RECIPIENT'S ACCESSION NO.
PB8 4 229723

4. TITLE AND SUBTITLE
Application of an Analysis Protocol to Identify Organic Compounds Not Identified by Spectrum Matching, Part 2: Appendices

5. REPORT DATE
August 1984

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
Joan T. Bursey

8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Research Triangle Institute
Research Triangle Park, NC 27709

10. PROGRAM ELEMENT NO.
CBHB1C

11. CONTRACT/GRANT NO.
68-03-2867

12. SPONSORING AGENCY NAME AND ADDRESS
U.S. Environmental Protection Agency--Athens GA
Office of Research and Development
Environmental Research Laboratory
Athens, GA 30613

13. TYPE OF REPORT AND PERIOD COVERED
Final, 12/82-2/84

14. SPONSORING AGENCY CODE
EPA/600/01

15. SUPPLEMENTARY NOTES
Part 1: Text

16. ABSTRACT

Industrial wastewater survey samples were analyzed for organic compounds not identified by spectrum matching. Analysis of the samples proceeded from an initial packed column GC/MS analysis for Priority Pollutants, through computerized spectrum matching for other compounds, to the present capillary column GC/MS analysis of a chosen set of sample extracts. Attention was focused on the spectra seen to occur frequently, yet not tentatively identified by spectrum matching. A plan for systematic study of these sample components was devised that included, in step-wise fashion, the use of high resolution gas chromatography, high resolution mass spectrometry, chemical ionization mass spectrometry with positive and negative ion detection, and Fourier transform infrared spectroscopy. Sample cleanup was used at all levels to mitigate interference. For 55 extracts in which components of interest were observed, accurate mass measurement was successfully used to generate chemical formulas in 35 cases. Of these, the results of 16 could be narrowed to one of two possibilities each. Tentative structures were proposed in six cases. Conclusions were: (1) that this type of compound identification effort is very time and labor intensive, (2) that labor costs are high because highly trained and experienced personnel are required, and (3) that the amount of definitive information that can be obtained by application of any of the analytical techniques discussed above ranges from minimal to extensive but that integration of information often is a complex problem.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT
RELEASE TO PUBLIC

19. SECURITY CLASS *(This Report)*
UNCLASSIFIED

20. SECURITY CLASS *(This page)*
UNCLASSIFIED

21. NO. OF PAGES
265

22. PRICE

EPA Form 2220-1 (9-73)

DISCLAIMER

The information in this document has been funded wholly or in part by the United States Environmental Protection Agency under Contract No. 68-03-2867 to Research Triangle Institute. It has been subject to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the U.S. Environmental Protection Agency.

FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure specific pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of environmental contaminants, the Analytical Chemistry Branch characterizes chemical constituents of water and soil.

Studies at this and other laboratories have shown that most compounds in survey analyses are not contained in present-day libraries of reference spectra. The abulity to identify the most frequently occurring of these compounds requires the application of high resolution mass spectrometry, Fourier-transform infrared spectroscopy, and other sophisticated analysis techniques. This report describes the application of a protocol for analyzing organics that are not represented in spectral libraries using effluent extracts from a variety of industries.

William T. Donaldson
Acting Director
Environmental Research Laboratory
Athens, Georgia

ABSTRACT

Industrial wastewater survey samples were analyzed for organic compounds not identified by spectrum matching. Analysis of the samples proceeded from an initial packed column GC/MS analysis for Priority Pollutants, through computerized spectrum matching for other organic compounds, to the present capillary column GC/MS analysis of a chosen set of sample extracts. Attention was focused on the spectra seen to occur frequently, yet not tentatively identified by spectrum matching. A plan for systematic study of these sample components was devised that included, in step-wise fashion, the use of high resolution gas chromatography (HRGC), high resolution mass spectrometry (HRMS), chemical ionization mass spectrometry (CIMS) with positive and negative ion detection (NCI), and Fourier transform infrared spectroscopy (FT-IR). Sample cleanup was used at all levels to mitigate interference. For 55 extracts in which components of interest were observed, accurate mass measurement was successfully used to generate chemical formulas in 35 cases. Of these, the results for 16 could be narrowed to one or two possibilities each. Tentative structures were proposed in six cases. Since the proposed compounds were not commercially available and the costs of synthesis were prohibitive, no further confirmation was made.

Conclusions were: (1) that this type of compound isolation/identification effort is very time and labor intensive, (2) that labor costs are high because highly trained and experienced personnel are required, and (3) that the amount of definitive information that can be obtained by application of any one of the analytical techniques discussed above (or of several of the techniques in succession) ranges from minimal to very high, but integration of all the information available is often not as simple as the analyst might wish.

This report was submitted in fulfillment of Contract Number 68-03-2867 by the Research Triangle Institute under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from December 1982 to February 1984, and work was completed as of February 1984.

Appendix A Figures

Number		Page
A-1	Extract 663 (acid), pharmaceuticals industry. Frequency of occurrence: 12	1
A-2	Extract 958 (b/n), paint and ink industry. Frequency of occurrence: 18	2
A-3	Extract 961 (b/n), paint and ink industry. Frequency of occurrence: 16	3
A-4	Extract 1135 (acid), pulp and paper industry. Frequency of occurrence: 7	4
A-5	Extract 1135 (acid), pulp and paper industry. Frequency of occurrence: 11	5
A-6	Extract 1222 (b/n), rum industry. Frequency of occurrence: 6	6
A-7	Extract 1235 (b/n), rum industry. Frequency of occurrence: 15	7
A-8	Extract 1234 (b/n), rum industry. Frequency of occurrence: 16	8
A-9	Extract 1630 (b/n), publicly owned treatment works. Frequency of occurrence: 5	9
A-10	Extract 1691 (acid), publicly owned treatment works. Frequency of occurrence: 10	10
A-11	Extract 1699 (b/n), publicly owned treatment works. Frequency of occurrence: 5	11
A-12	Extract 1733 (b/n), leather tanning industry. Frequency of occurrence: N/A	12
A-13	Extract 2373 (acid), inorganic chemicals industry. Frequency of occurrence: 21	13
A-14	Extract 2373 (acid), inorganic chemicals industry. Frequency of occurrence: 10	14

Appendix A Figures (Cont'd)

Number Page

A-15 Extract 2965 (acid), publicly owned treatment works. Frequency of occurrence: 5 15

A-16 Extract 2981 (b/n), pulp and paper industry. Frequency of occurrence: N/A 16

A-17 Extract 4050 (acid), pulp and paper industry. Frequency of occurrence: 6 17

A-18 Extract 4050 (acid), pulp and paper industry. Frequency of occurrence: 6 18

A-19 Extract 4064 (acid), pulp and paper industry. Frequency of occurrence: 9 19

A-20 Extract 4139 (b/n), pesticides manufacturing industry. Frequency of occurrence: 5 20

A-21 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 6 21

A-22 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 6 22

A-23 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 5 23

A-24 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 5 24

A-25 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 9 25

A-26 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 22 26

A-27 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 6 27

A-28 Extract 4748 (b/n), electronics industry. Frequency of occurrence: 6 28

A-29 Extract 5152 (b/n), mechanical products industry. Frequency of occurrence: 15 29

A-30 Extract 5205 (b/n), coal mining industry. Frequency of occurrence: 15 30

Appendix A Figures (Cont'd)

Number Page

A-31 Extract 5207 (b/n), coal mining industry. Frequency of occurrence: 17 31

A-32 Extract 5424 (acid), mechanical products industry. Frequency of occurrence: 35 32

A-33 Extract 5425 (acid), mechanical products industry. Frequency of occurrence: 22 33

A-34 Extract 5671 (acid), printing and publishing industry. Frequency of occurrence: 15 34

A-35 Extract 5969 (b/n), publicly owned treatment works. Frequency of occurrence: 47 35

A-36 Extract 7979 (b/n), petroleum refining industry. Frequency of occurrence: 5 36

A-37 Extract 8218 (acid), timber products industry. Frequency of occurrence: 9 37

A-38 Extract 8283 (b/n), timber products industry. Frequency of occurrence: N/A 38

A-39 Extract 8320 (b/n), timber products industry. Frequency of occurrence: 58 39

A-40 Extract 8751 (b/n), ore mining industry. Frequency of occurrence: 5 40

A-41 Extract 8789 (acid), ore mining industry. Frequency of occurrence: N/A 41

A-42 Extract 8996 (acid), publicly owned treatment works. Frequency of occurrence: 37 42

A-43 Extract 9275 (b/n), mechanical products industry. Frequency of occurrence: 6 43

A-44 Extract 9284 (b/n), mechanical products industry. Frequency of occurrence: 10 44

A-45 Extract 9381 (b/n), soaps and detergents industry. Frequency of occurrence: N/A 45

A-46 Extract 9914 (b/n), inorganic chemicals industry. Frequency of occurrence: 12 46

Appendix A Figures (Cont'd)

Number Page

A-47 Extract 13158 (b/n), publicly owned treatment works. Frequency of occurrence: N/A 47

A-48 Extract 13614 (acid), iron and steel manufacturing industry. Frequency of occurrence: 14 48

A-49 Extract 13738 (b/n), publicly owned treatment works. Frequency of occurrence: 16 49

A-50 Extract 14615 (b/n), organic chemicals industry. Frequency of occurrence: 8 50

A-51 Extract 15850 (b/n), organics and plastics industry. Frequency of occurrence: N/A 51

A-52 Extract 16610 (b/n), synfuels industry. Frequency of occurrence: 30 52

A-53 Extract 17897 (b/n), organics and plastics industry. Frequency of occurrence: N/A 53

A-54 Extract 19245 (acid), electronics industry. Frequency of occurrence: 8 54

A-55 Extract 19245 (acid), electronics industry. Frequency of occurrence: 9 55

Appendix B Figures

Number		Page
B-1	Packed column chromatogram, extract 1634 (acid), publicly owned treatment works	56
B-2	Extract 1634 (acid), publicly owned treatment works. Frequency of occurrence: 13	57
B-3	Capillary chromatogram of extract 1634 (acid), publicly owned treatment works	58
B-4	Mass spectrum of EPA compound of interest, extract 1634 (acid), publicly owned treatment works, scan #845	59
B-5	Conclusions	60
B-6	Packed column chromatogram of extract 1678 (acid), publicly owned treatment works	61
B-7	Extract 1678 (acid), publicly owned treatment works. Frequency of occurrence: 7	62
B-8	Capillary chromatogram, extract 1678 (acid), publicly owned treatment works	63
B-9	Mass spectrum of EPA compound of interest, extract 1678 (acid), publicly owned treatment works, scan #355	64
B-10	Conclusions	65
B-11	Packed column chromatogram, extract 2389 (acid), inorganic chemicals industry	66
B-12	Extract 2389 (acid), inorganic chemicals industry. Frequency of occurrence: N/A	67
B-13	Capillary chromatogram, extract 2389 (acid), inorganic chemicals industry	68
B-14	Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #1834	69

Appendix B Figures (Cont'd)

Number Page

B-15 Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemical industry, scan #2179 70

B-16 Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2559 71

B-17 Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2592 72

B-18 Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2703 73

B-19 Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2729 74

B-20 Conclusions 75

B-21 Packed column chromatogram, extract 2390 (acid), inorganic chemicals industry 76

B-22 Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 7 77

B-23 Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 6 78

B-24 Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 9 79

B-25 Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 9 80

B-26 Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 5 81

B-27 Capillary chromatogram, extract 2390 (acid), inorganic chemicals industry 82

B-28 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, not identified, scan #758 83

B-29 Mass spectrum of EPA compound of interest, one possible isomer; extract 2390 (acid), inorganic chemicals industry, scan #488 84

Appendix B Figures (Cont'd)

Number Page

B-30 Mass spectrum of EPA compound of interest, one possible isomer; extract 2390 (acid), inorganic chemicals industry, scan #806 85

B-31 Mass spectrum of EPA compound of interest, one possible isomer; extract 2390 (acid), inorganic chemicals industry, scan #1147 86

B-32 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #861 87

B-33 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #1079 88

B-34 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, scan #1025 89

B-35 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #668 90

B-36 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #684 91

B-37 Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #793 92

B-38 Other halogenated compound observed in extract 2390 (acid) inorganic chemicals industry, spectrum unidentified, scan #354 93

B-39 Other halogenated compound observed in extract 2389 (acid), inorganic chemicals industry, spectrum unidentified, scan #549 94

B-40 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #599 95

B-41 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #685 96

Appendix B Figures (Cont'd)

Number Page

B-42 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #794 97

B-43 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #806 98

B-44 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #877 99

B-45 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #955 100

B-46 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals indusrry, spectrum unidentified, scan #1005 101

B-47 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1025 102

B-48 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1042 103

B-49 Other halogenated compound observed in extract 2390 (acid), inortanic chemicals industry, spectrum unidentified, scan #1080 104

B-50 Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1147 105

B-51 Table of compounds identified and tentatively identified . . 106

B-52 Conclusions 107

B-53 Packed column chromatogram, extract 3005 (base/neutral), porcelain/enameling industry 108

B-54 Extract 3005 (b/n), porcelain/enameling industry. Frequency of occurrence: 27 109

Appendix B Figures (Cont'd)

Number Page

B-55 Packed column chromatogram of extract 2016 (b/n), pulp and paper industry. (NOTE: This extract was actually supplied and analyzed rather than extract 3006 (b/n) . . . 110

B-56 Extract 2006 (b/n), porcelain/enameling industry. Frequency of occurrence: 27 111

B-57 Capillary chromatogram, extract 3005 (b/n), porcelain/enameling industry 112

B-58 EPA compound of interest, extract 3005 (b/n), porcelain/enameling industry, scan #1113 113

B-59 Capillary chromatogram, extract 3016 (b/n), pulp and paper industry, scan #1117 114

B-60 Mass spectrum, EPA compound of interest, extract 2016 (b/n), pulp and paper industry, scan #1120 115

B-61 Conclusions 116

B-62 Packed column chromatogram, extract 4400 (b/n), auto and other laundries industry 117

B-63 Extract 4400 (b/n), auto and other laundries industry. Frequency of occurrence: 15 118

B-64 Capillary chromatogram, extract 4400 (b/n), auto and other laundries industry 119

B-65 EPA compound of interest, extract 4400 (b/n), auto and other laundries industry, one isomer, scan #1878 120

B-66 EPA compound of interest, extract 4400 (b/n), auto and other laundries industry, one isomer, scan #1927 121

B-67 Conclusions 122

B-68 Packed column chromatogram, extract 4752 (b/n), electronics industry 123

B-69 Extract 4752 (b/n), electronics industry. Frequency of occurrence: N/A 124

Appendix B Figures (Cont'd)

Numbers Page

B-70 Capillary chromatogram, extract 4752 (b/n), electronics industry 125

B-71 EPA compound of interest, extract 4752 (b/n), electronics industry 126

B-72 Conclusions 127

B-73 Packed column chromatogram, extract 4809 (acid), porcelain/enameling industry 128

B-74 Extract 4809 (acid), porcelain/enameling industry. Frequency of occurrence: 15 129

B-75 Capillary chromatogram, extract 4809 (acid), porcelain/enameling industry 130

B-76 EPA compound of interest, extract 4809 (acid), porcelain/enameling industry, scan #2190 131

B-77 Conclusions 132

B-78 Packed column chromatogram, extract 5694 (b/n), organic chemicals industry 133

B-79 Extract 5694 (b/n), organic chemicals industry. Frequency of occurrence: 17 134

B-80 Capillary chromatogram, extract 5694 (b/n), organic chemicals industry 135

B-81 Mass spectrum of EPA compound of interest, extract 5694 (b/n), organic chemicals industry, one isomer, scan #1961 136

B-82 Mass spectrum of EPA compound of interest, extract 5694 (b/n), orgainc chemicals industry, one isomer, scan #1985 137

B-83 Mass spectrum of EPA compound of interest, extract 5694 (b/n), organic chemicals industry, one isomer, scan #2065 138

B-84 Mass spectrum of EPA compound of interest, extract 5694 (b/n), organic chemicals industry, one isomer, scan #2029 139

B-85 Representative isobutane chemical ionization mass spectrum of EPA compound of interest, scan #2081 140

B-86 Conclusions 141

Appendix B Figures (Cont'd)

Number		Page
B-87	Packed column chromatogram, extract 5894 (acid), pesticides manufacturing industry	142
B-88	Extract 5894 (acid), pesticides manufacturing industry. Frequency of occurrence: 5	143
B-89	Capillary chromatogram, extract 5849 (acid), pesticides manufacturing industry	144
B-90	EPA compound of interest, extract 5894 (acid), pesticides manufacturing industry, scan #2190	145
B-91	Conclusions	146
B-92	Packed column chromatogram, extract 5859 (acid), pesticides manufacturing industry	147
B-93	Extract 5859 (acid), pesticides manufacturing industry. Frequency of occurrence: 7	148
B-94	Capillary chromatogram, extract 5859 (acid), pesticides manufacturing industry	149
B-95	Mass spectrum of EPA compound of interest, extract 5859 (acid), pesticides manufacturing industry, scan #2771 . .	150
B-96	Conclusions	151
B-97	Packed column chromatogram, extract 6057 (acid), publicly owned treatment works	152
B-98	Extract 6057 (acid), publicly owned treatment works. Frequency of occurrence: 6	153
B-99	Capillary chromatogram, extract 6057 (acid), publicly owned treatment works	154
B-100	Mass spectrum of EPA compound of interest, extract 6057 (acid), publicly owned treatment works, one isomer, scan #1507	155
B-101	Mass spectrum of EPA compound of interest, extract 6057 (acid), publicly owned treatment works, one isomer, scan #1645	156
B-102	Conclusions	157

Appendix B Figures (Cont'd)

Number Page

B-103 Packed column chromatogram, extract 8289 (b/n), timber products industry 158

B-104 Extract 8289 (b/n), timber products industry. Frequency of occurrence: 5 159

B-105 Capillary chromatogram, extract 8289 (b/n), timber products industry 160

B-106 Mass spectrum of EPA compound of interest, extract 8289 (b/n), timber products industry, scan #830 161

B-107 Conclusions 162

B-108 Packed column chromatogram, extract 11722 (acid), rubber processing industry 163

B-109 Capillary chromatogram, extract 11722 (acid), rubber processing industry 164

B-110 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #261 165

B-111 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #387 166

B-112 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #430 167

B-113 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #486 168

B-114 Mass spectrum of unidentified brominated constiturent, extract 11722 (acid), rubber processing industry, scan #617 169

B-115 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #821 170

Appendix B Figures (Cont'd)

Number Page

B-116 Mass spectrum of unidentifed brominated constituent, extract 11722 (acid), rubber processing industry, scan #829 171

B-117 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #994 172

B-118 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1604 173

B-119 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1025 174

B-120 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1202 175

B-121 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1058 176

B-122 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1130 177

B-123 Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1442 178

B-124 Tentatively identified compounds 179

B-125 Conclusions 180

B-126 Packed column chromatogram, extract 11939 (b/n), timber products industry 181

B-127 Extract 11939 (b/n), timber products industry 182

B-128 Capillary chromatogram, extract 11939 (b/n), timber products industry 183

B-129 EPA compound of interest, extract 11939 (b/n), timber products industry 184

Appendix B Figures (Cont'd)

Number Page

B-130 Capillary chromatogram (chemical ionization mass spectrometry), extract 11939 (b/n), timber products industry. Note lack of correspondence to EI profile 185

B-131 Chemical ionization mass spectrum, same retention time as EPA compound of interest, scan #2308 186

B-132 Conclusions 187

B-133 Packed column chromatogram, extract 16098 (acid), publicly owned treatment works 188

B-134 Extract 16098 (acid), publicly owned treatment works. Frequency of occurrence: 24 189

B-135 Capillary chromatogram, extract 16098 (acid), publicly owned treatment works 190

B-136 EPA compound of interest, extract 16098 (acid), publicly owned treatment works 191

B-137 Computerized library search, EPA compound of interest, extract 16098 (acid), publicly owned treatment works . . . 192

B-138 Paced column chromatogram, extract 16100 (acid), publicly owned treatment works 193

B-139 Extract 16100 (acid), publicly owned treatment works. Frequency of occurrence: 24 194

B-140 Capillary chromatogram, extract 16100 (acid), publicly owned treatment works 195

B-141 Mass spectrum of EPA compound of interest, extract 16100 (acid), publicly owned treatment works, scan #2060 196

B-142 Conclusions 197

B-143 Packed column chromatogram, extract 16217 (b/n), publicly owned treatment works 198

B-144 Extract 16217 (b/n), publicly owned treatment works. Frequency of occurrence: 11 199

B-145 Capillary chromatogram, extract 16217 (b/n), publicly owned treatment works 200

Appendix B Figures (Cont'd)

Number Page

B-146 Mass spectrum, EPA compound of interest, extract 16217 (b/n), publicly owned treatment works 201

B-147 Packed column chromatogram, extract 17245 (b/n), publicly owned treatment works 202

B-148 Extract 17245 (b/n), publicly owned treatment works. Frequency of occurrence: 11 203

B-149 Capillary chromatogram, extract 17245 (b/n), publicly owned treatment works 204

B-150 Mass spectrum of EPA compound of interest, extract 16245 (b/n), publicly owned treatment works, scan #1677 205

B-151 Packed column chromatogram, extract 19010 (b/n), pharmaceuticals industry 206

B-152 Extract 19010 (b/n), pharmaceuticals industry. Frequency of occurrence: 11 207

B-153 Capillary chromatogram, extract 19010 (b/n), pharmaceuticals industry 208

B-154 Mass spectrum of EPA compound of interest, extract 19010 (b/n), pharmaceuticals industry 209

B-155 Conclusions 210

B-156 Packed column chromatogram, extract 16683 (acid), synfuels industry 211

B-157 Extract 16683 (acid), synfuels industry. Frequency of occurrence: 12 212

B-158 Capillary chromatogram, extract 16683 (acid), synfuels industry 213

B-159 Mass spectrum of EPA compound of interest, extract 16683 (acid), synfuels industry, scan #4996 214

B-160 Packed column chromatogram, extract 15700 (acid), pulp and paper industry 215

B-161 Extract 15700 (acid), pulp and paper industry. Frequency of occurrence: 12 216

Appendix B Figures (Cont'd)

Number		Page
B-162	Capillary chromatogram, extract 15700 (acid), pulp and paper industry	217
B-163	Mass spectrum of EPA compound of interest, extract 15700 (acid), pulp and paper industry, scan #4939	218
B-164	Conclusions	219
B-165	Packed column chromatogram, extract 20339 (b/n), timber products industry	221
B-166	Extract 20339 (b/n), timber products industry. Frequency of occurrence: N/A	222
B-167	Capillary chromatogram, extract 20339 (b/n), timber products industry	223
B-168	Mass spectrum of EPA compound of interest, extract 20339 (b/n), timber products industry, scan #1834	224
B-169	Conclusions	225
B-170	Packed column chromatogram, extract 20435 (acid), publicly owned treatment works	226
B-171	Extract 20435 (acid), publicly owned treatment works. Frequency of occurrence: 33	227
B-172	Capillary chromatogram, extract 20435 (acid), publicly owned treatment works	228
B-173	Mass spectrum of EPA compound of interest, extract 20435 (acid), publicly owned treatment works, one isomer, scan #1646	229
B-174	Conclusions	230
B-175	Packed column chromatogram, extract 20437 (acid), publicly owned treatment works	231
B-176	Extract 20437 (acid), publicly owned treatment works. Frequency of occurrence: N/A	232
B-177	Capillary chromatogram, extract 20437 (acid), publicly owned treatment works	233

Appendix B Figures (Cont'd)

Number **Page**

B-178 Mass spectrum of EPA compound of interest, extract 20437 (acid), publicly owned treatment works, one isomer, scan #1737 234

B-179 Mass spectrum of EPA compound of interest, extract 20437 (acid), publicly owned treatment works, one isomer, scan #1697 235

B-180 Library search of EPA compound of interest, extract 20437 (acid), publicly owned treatment works 236

B-181 Packed column chromatogram, extract 20474 (acid), nonferrous metals industry 237

B-182 Extract 20474 (acid), nonferrous metals industry. Frequency of occurrence: N/A 238

B-183 Capillary chromatogram, extract 20474 (acid), nonferrous metals industry 239

B-184 Mass spectrum of EPA compound of interest, extract 20474 (acid), nonferrous metals industry, one isomer, scan #2478 240

B-185 Mass spectrum of EPA compound of interest, extract 20474 (acid0, nonferrous metals industry, one isomer, scan #2536 241

B-186 Conclusions 242

APPENDIX A: Mass Spectra of Components of Interest as Obtained from Computerized Analysis by U.S. Environmental Protection Agency

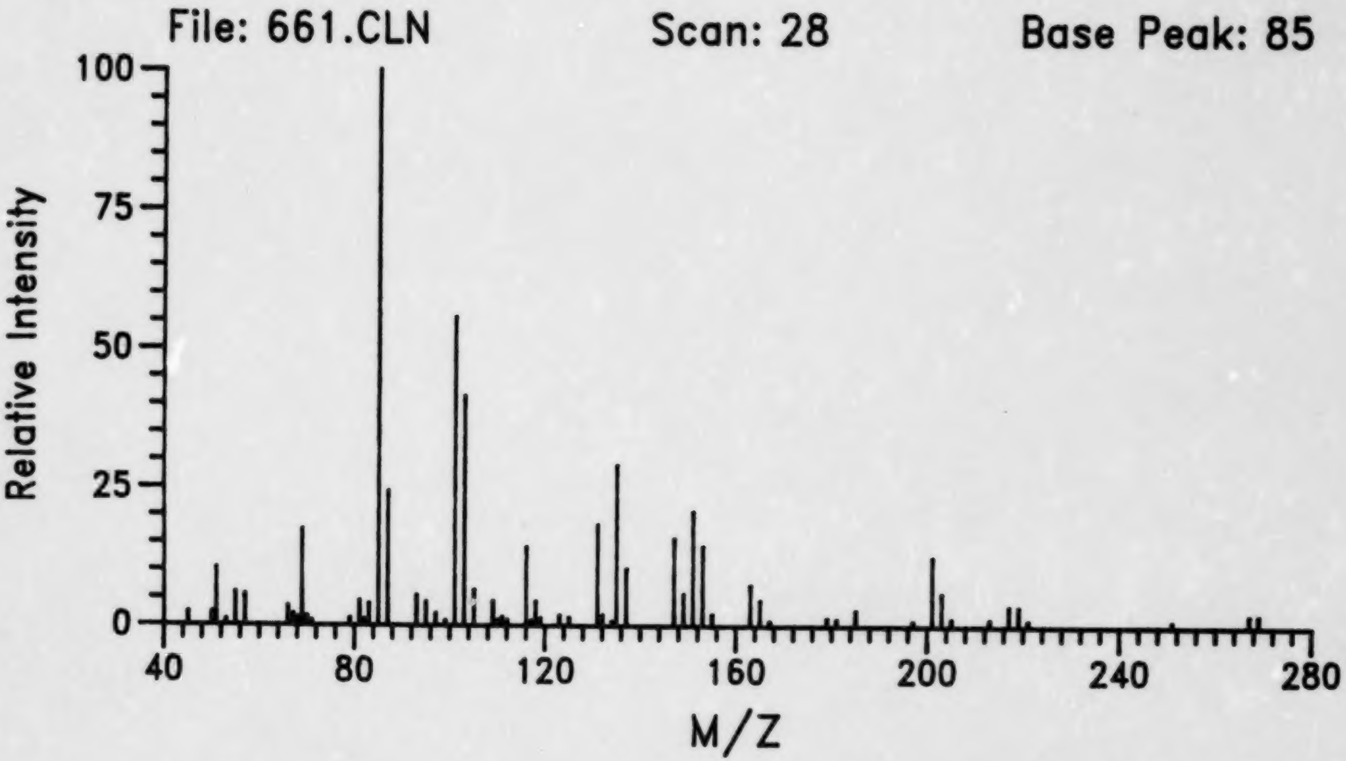


Figure A-1. Extract 663 (Acid), Pharmaceuticals Industry. Frequency of occurrence: 12.

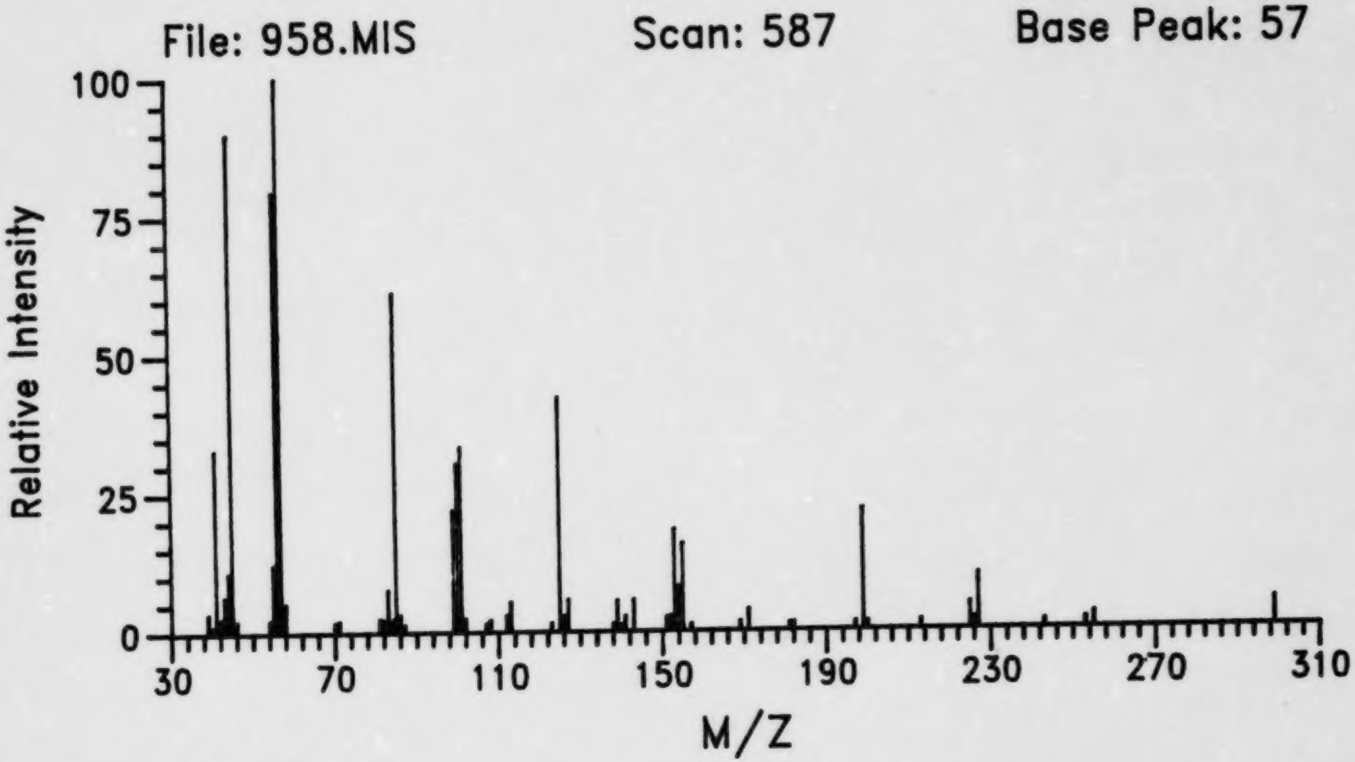


Figure A-2. Extract 958 (B/N), Paint and Ink Industry. Frequency of occurrence: 18.

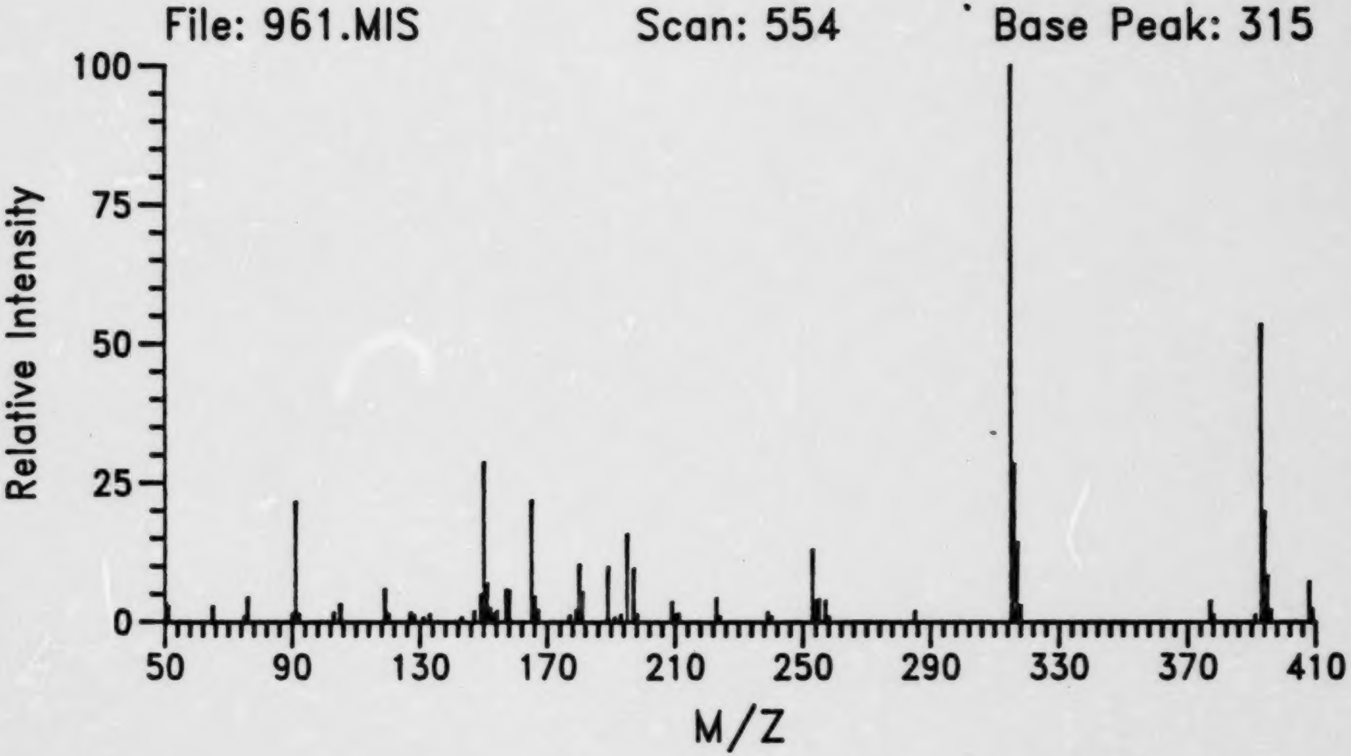


Figure A-3. Extract 961 (base/neutral), paint and ink industry. Frequency of occurrence: 16.

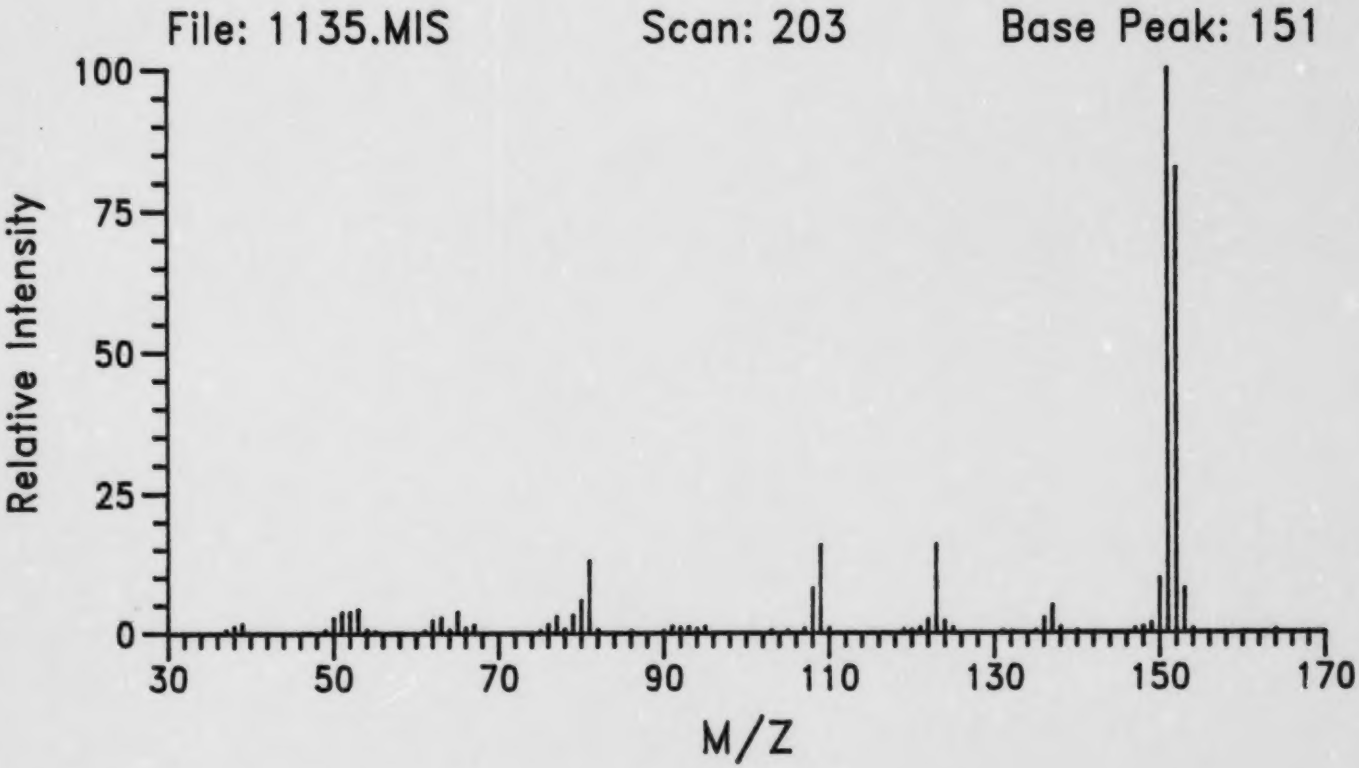


Figure A-4. Extract 1135 (acid), pulp and paper industry. Frequency of occurrence: 7.

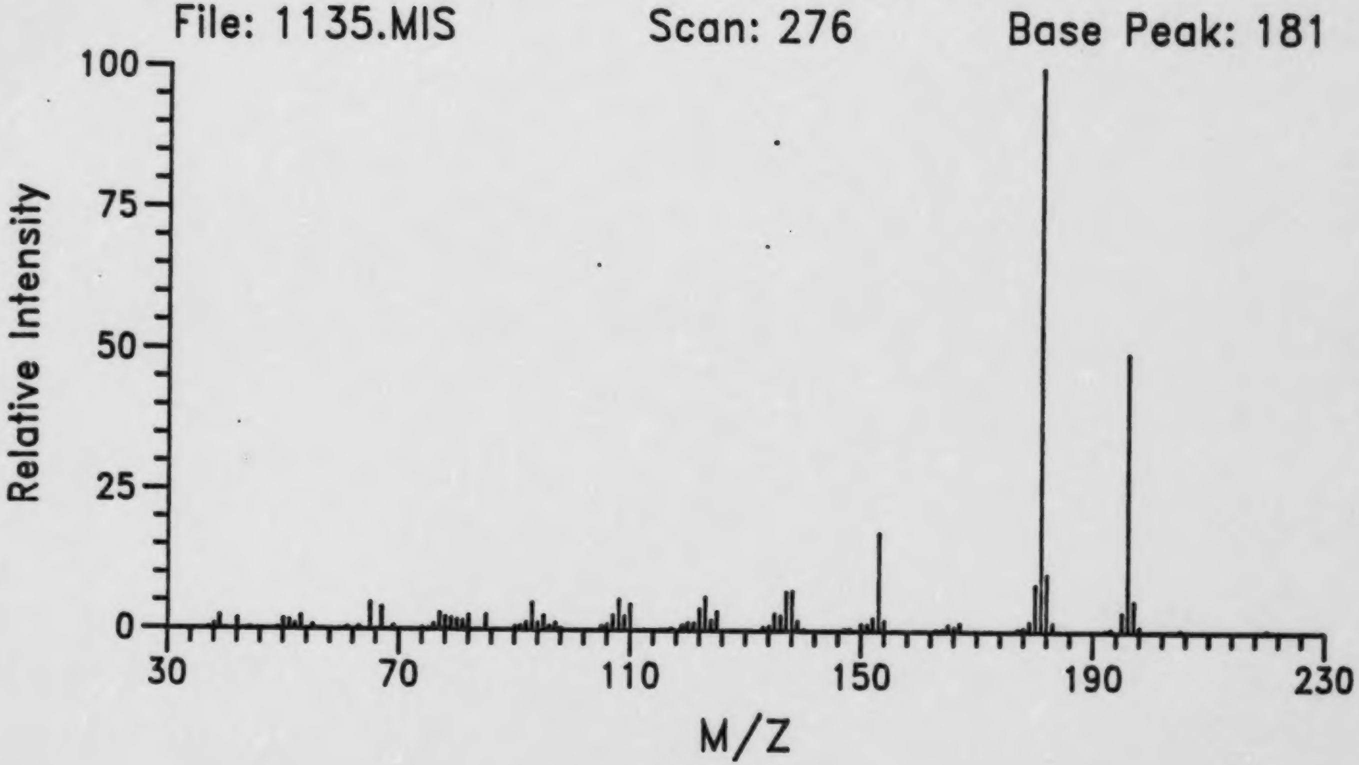


Figure A-5. Extract 1135 (acid), pulp and paper industry. Frequency of occurrence: 11.

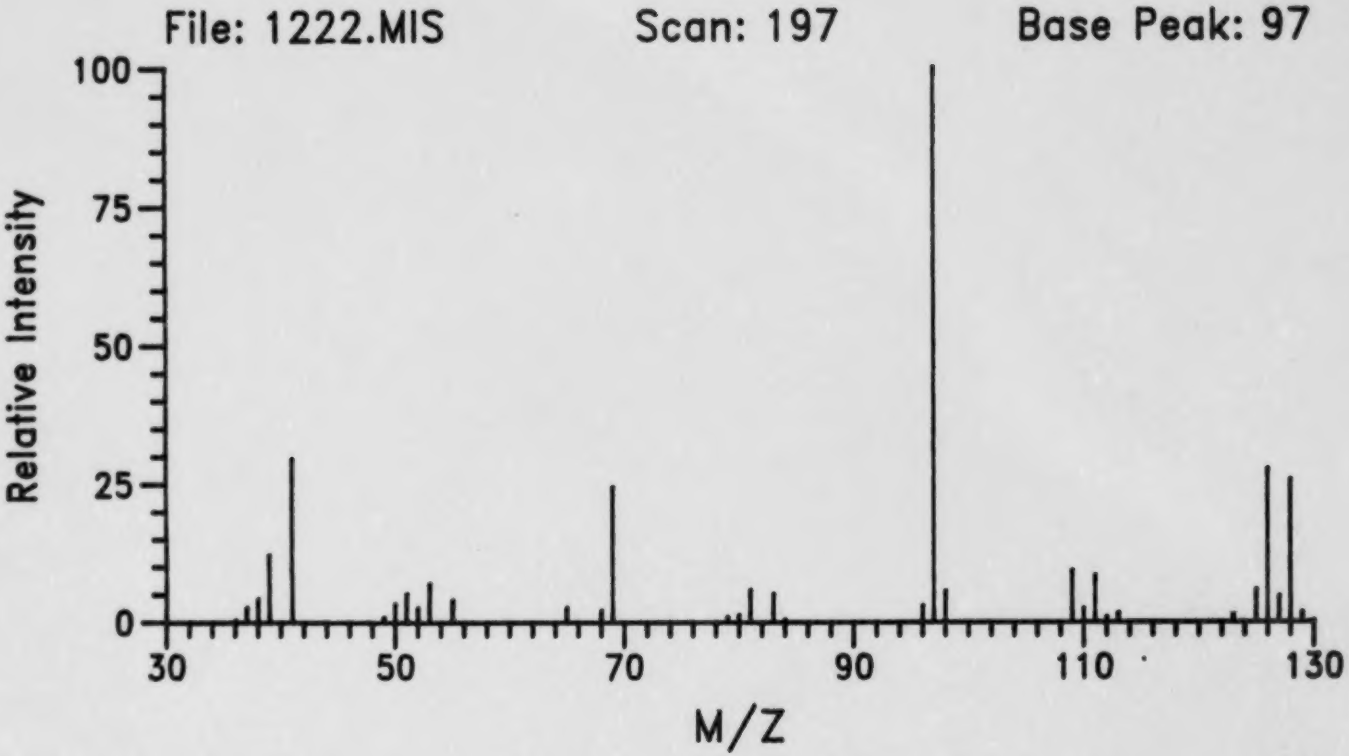


Figure A-6. Extract 1222 (Base/Neutral), Rum Industry. Frequency of occurrence: 6.

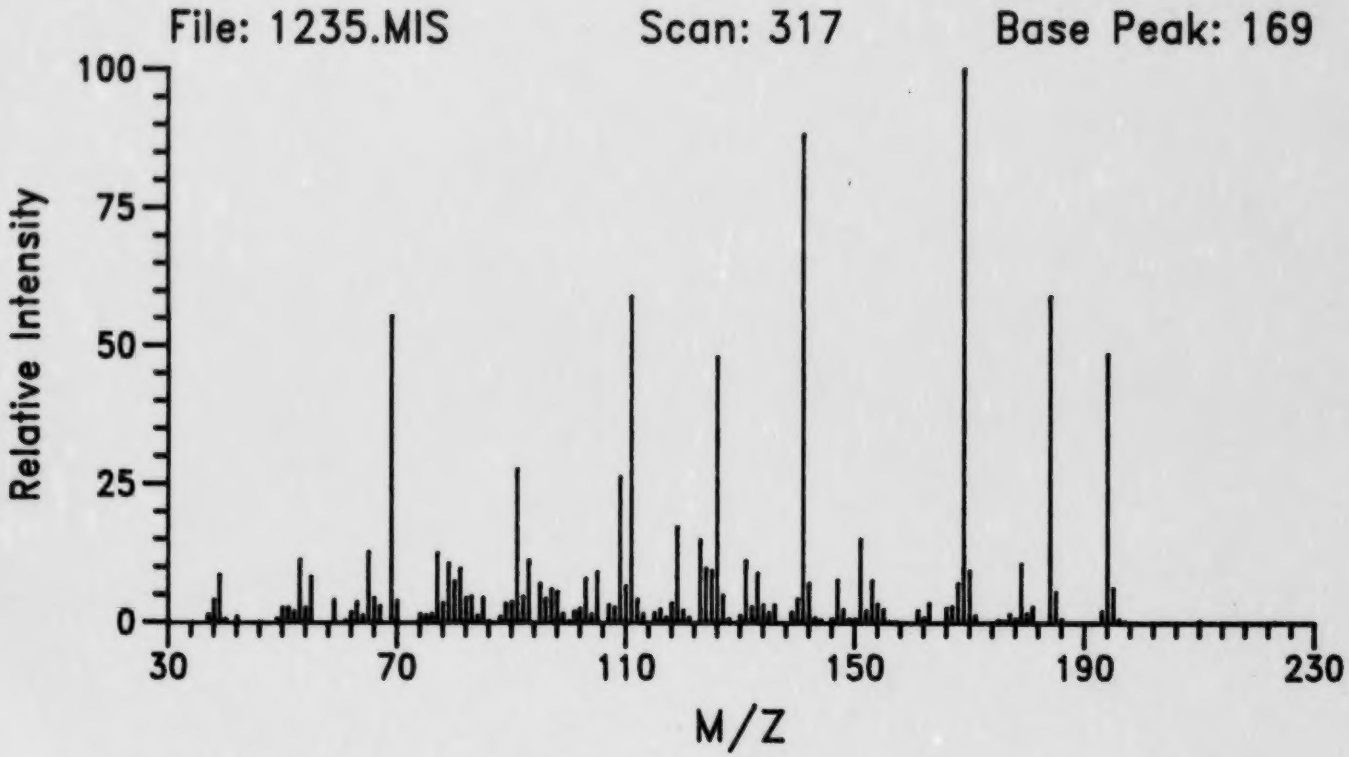


Figure A-7. Extract 1235 (Base/Neutral), Rum Industry. Frequency of occurrence: 15.

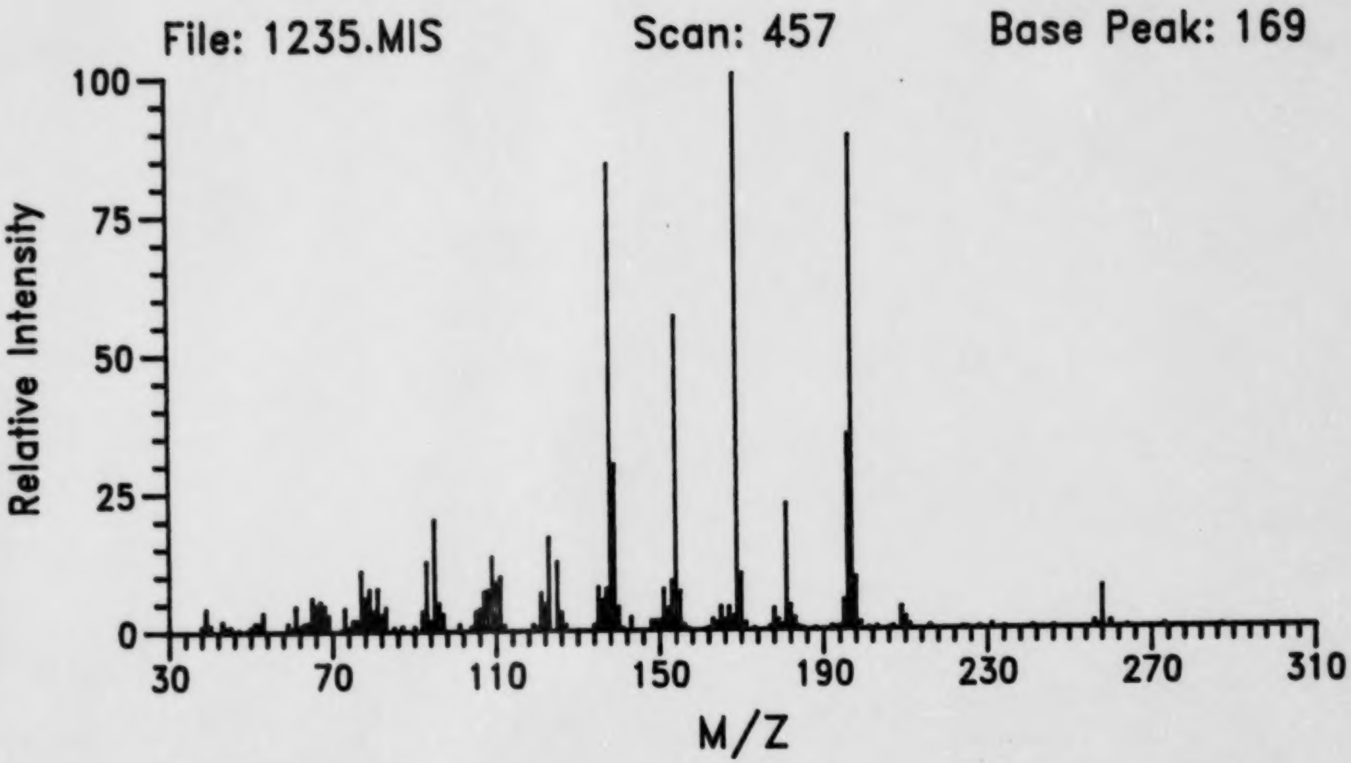


Figure A-8. Extract 1235 (Base/Neutral), Rum Industry. Frequency of occurrence: 16.

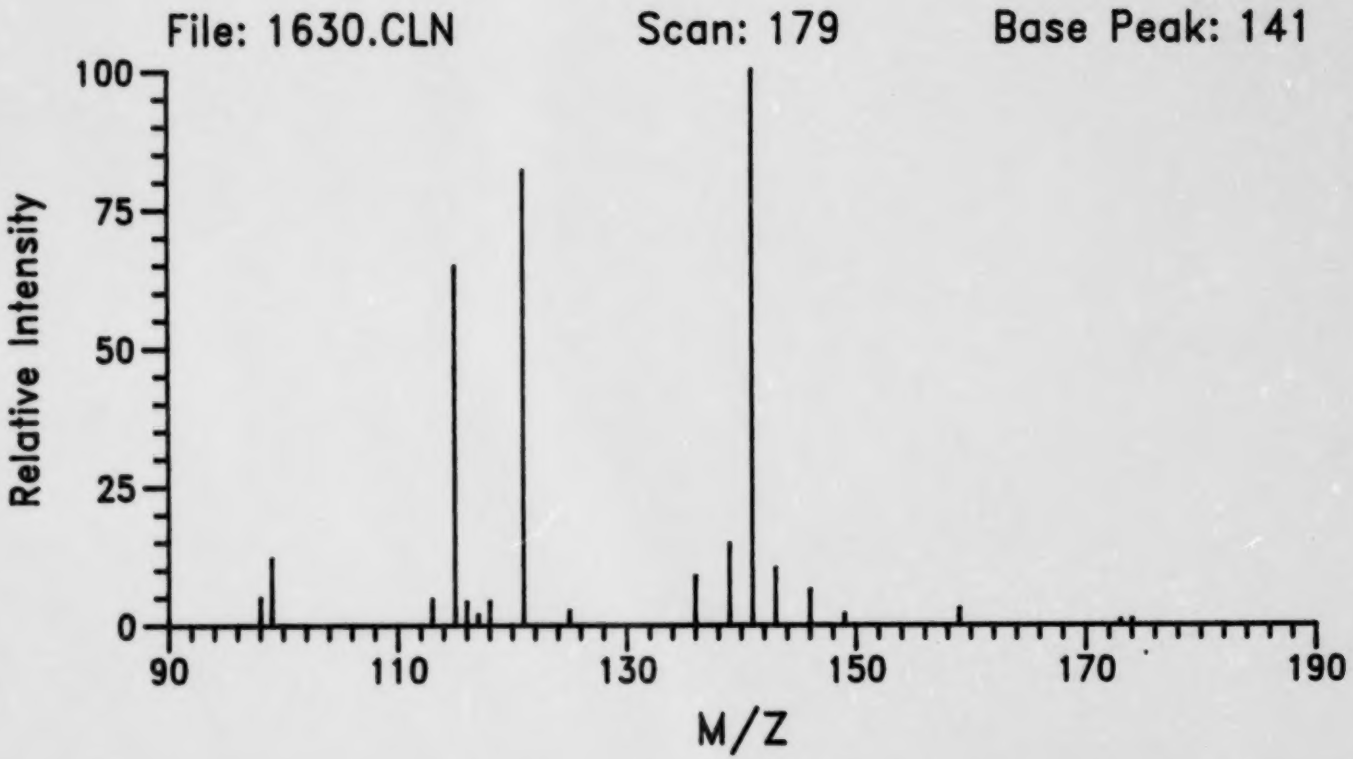


Figure A- 9. Extract 1630 (Base/Neutral), Publicly Owned Treatment Works. Frequency of occurrence: 5.

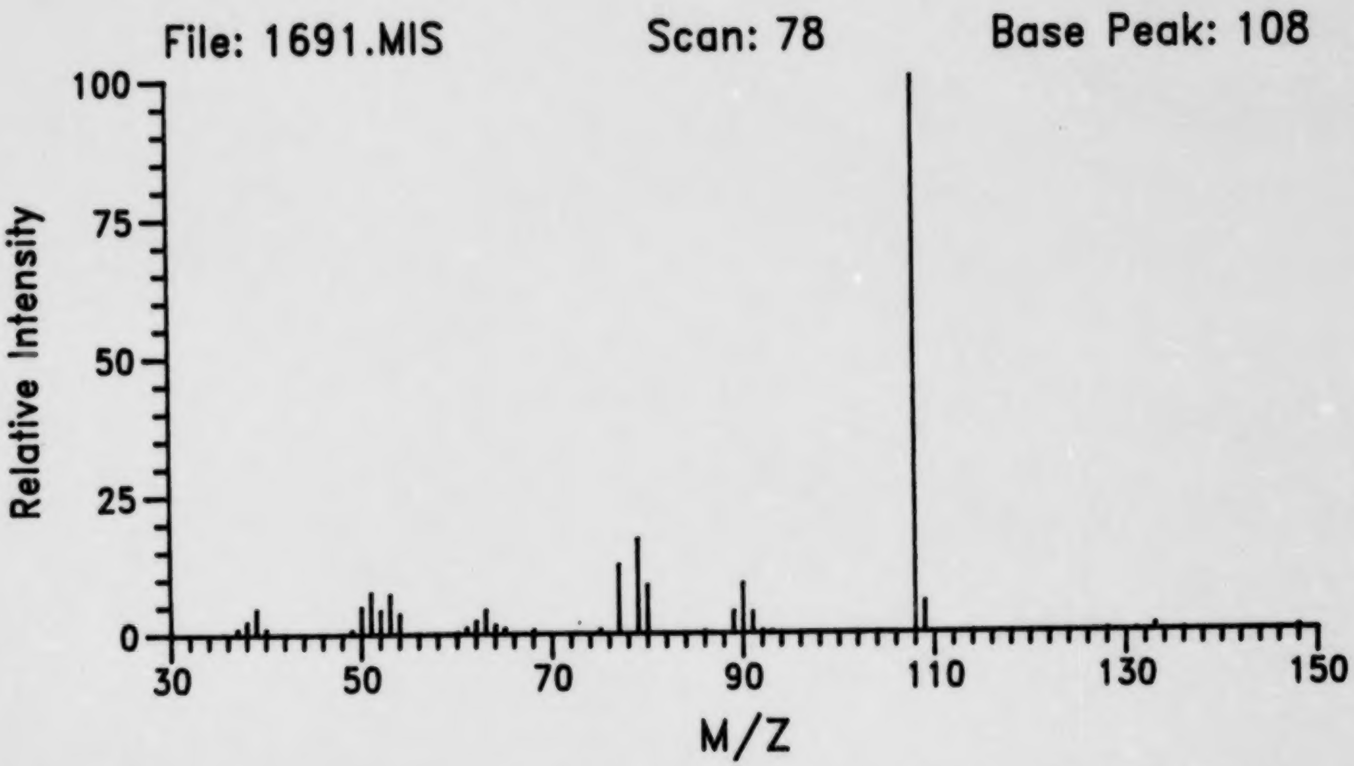


Figure A-10. Extract 1691 (Acid), Publicly Owned Treatment Works. Frequency of occurrence: 5.

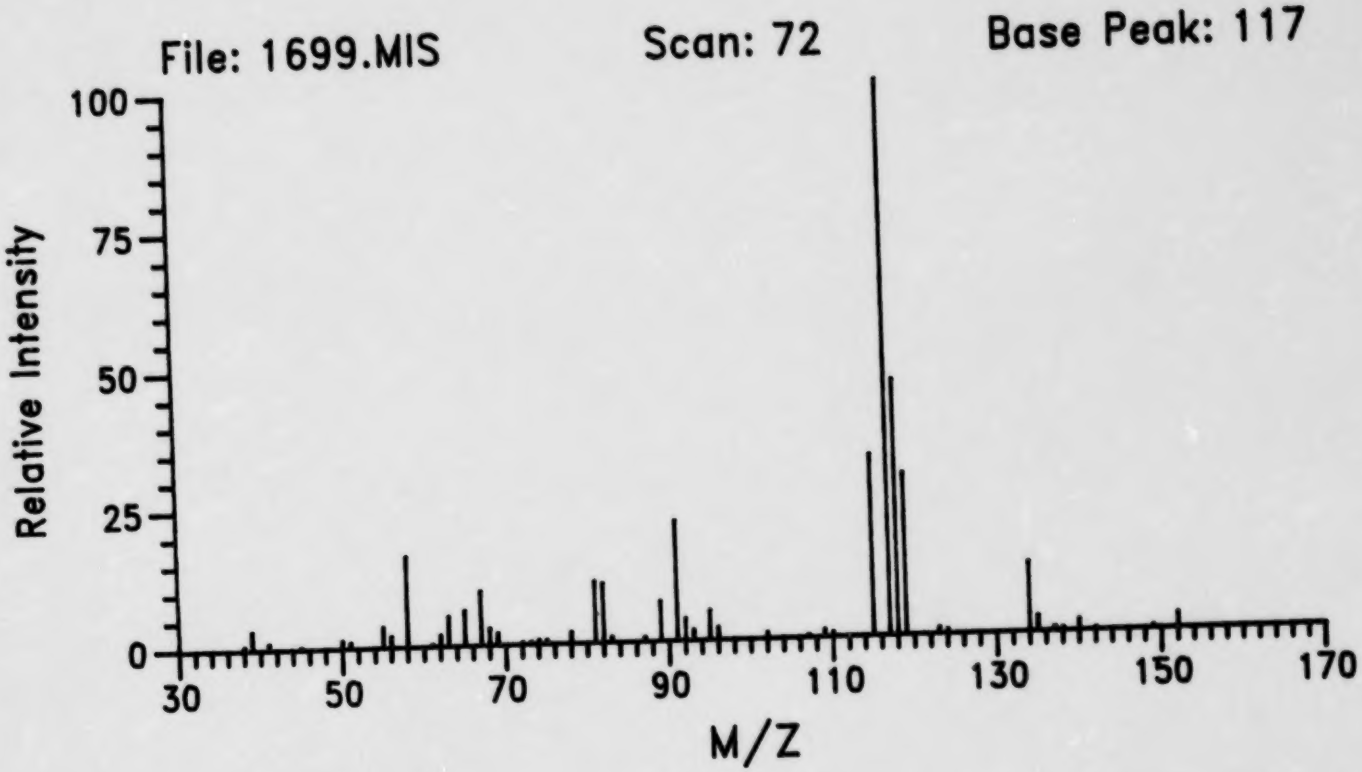


Figure A-11. Extract 1699 (Base/Neutral), Publicly Owned Treatment Works. Frequency of occurrence: 5.

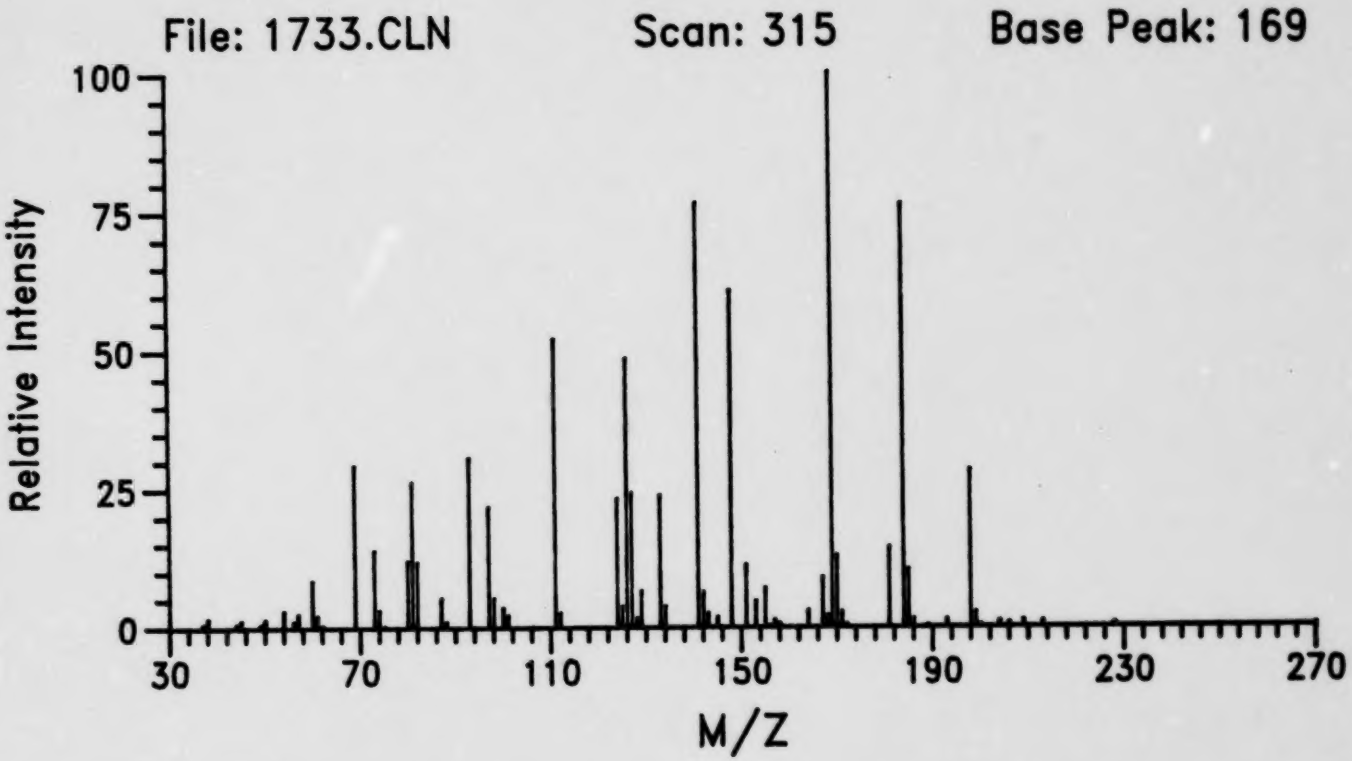


Figure A-12. Extract 2373 (acid), Inorganic Chemicals Industry. Frequency of occurrence: 21.

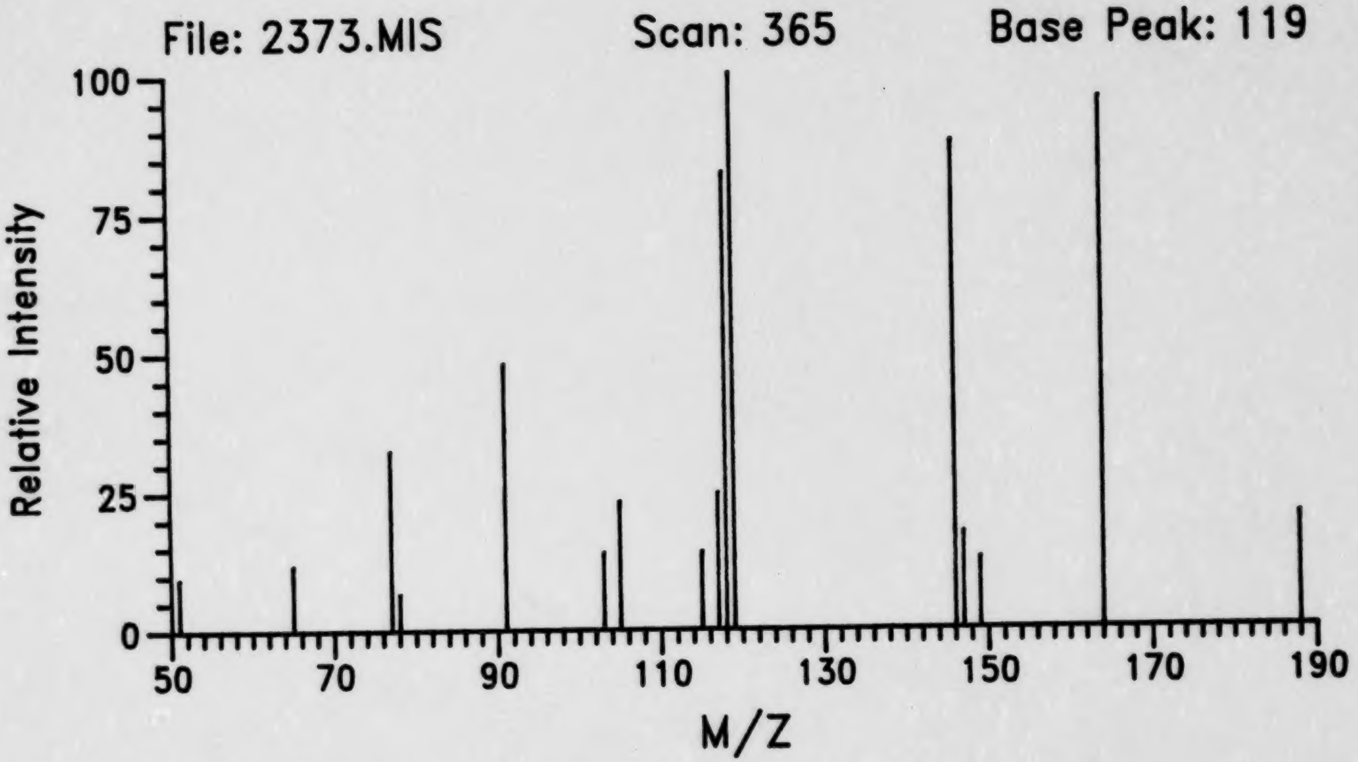


Figure A-13. Extract 2373 (Acid), Inorganic Chemicals Industry. Frequency of occurrence: 21.

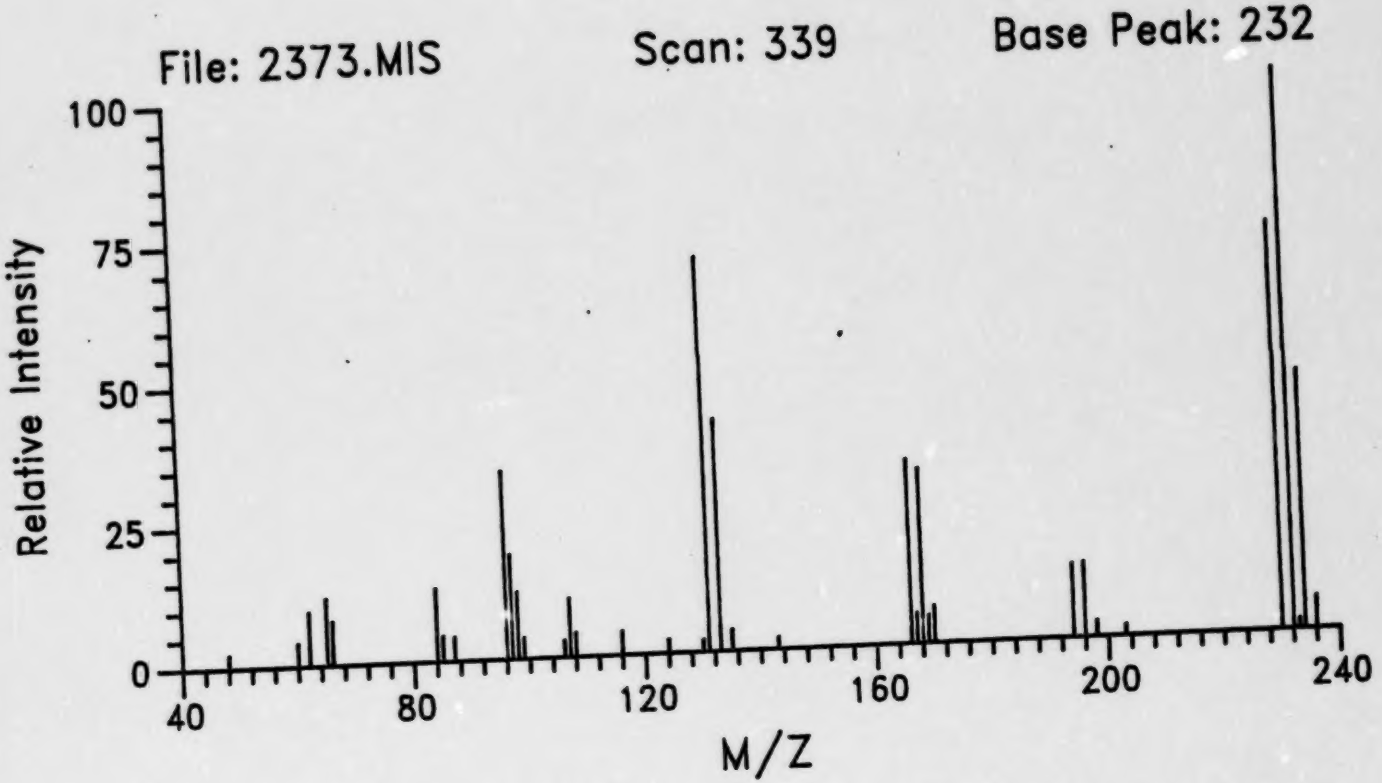


Figure A-14. Extract 2373 (Acid), Inorganic Chemicals Industry. Frequency of occurrence: 10.

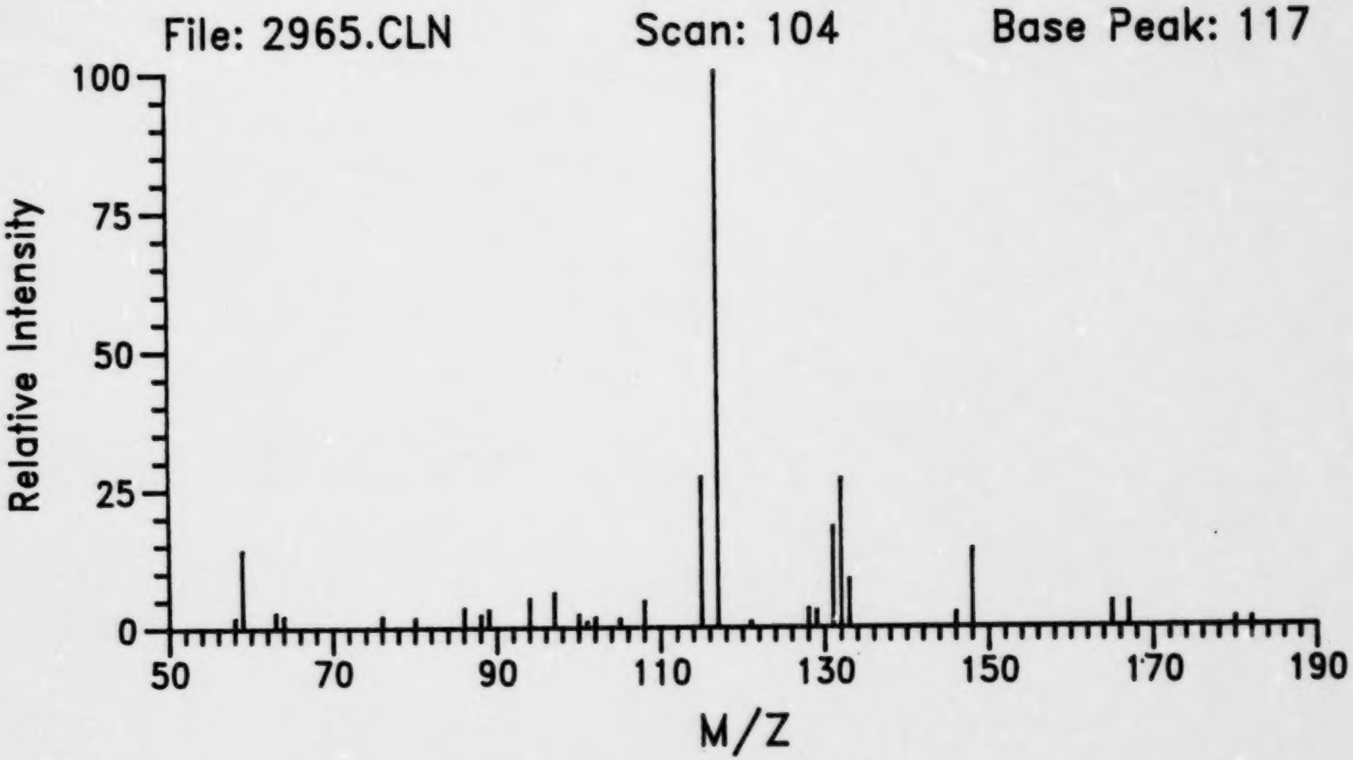


Figure A-15. Extract 2965 (Acid), Publicly Woned Treatment Works. Frequency of occurrence: 5.

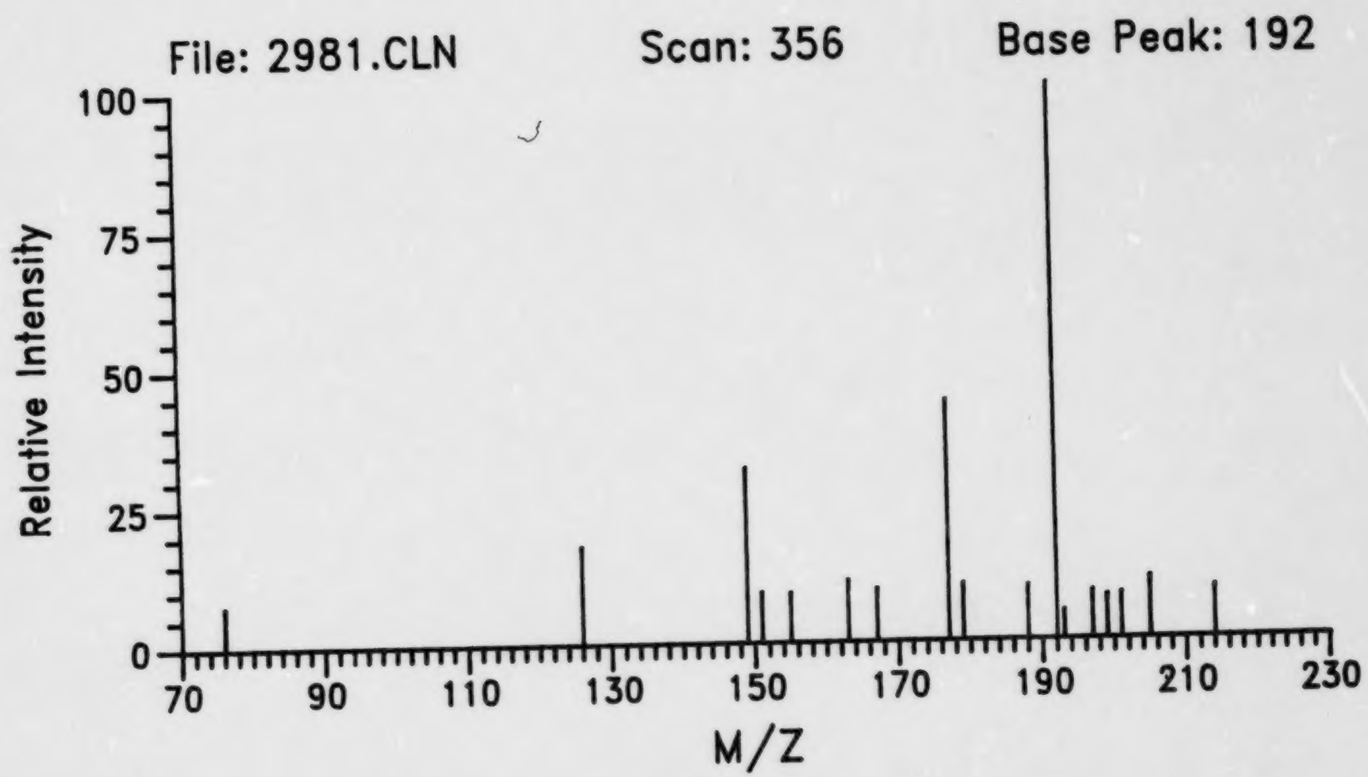


Figure A-16. Extract 2981 (Base/Neutral), Pulp and Paper Industry. Frequency of occurrence: N/A.

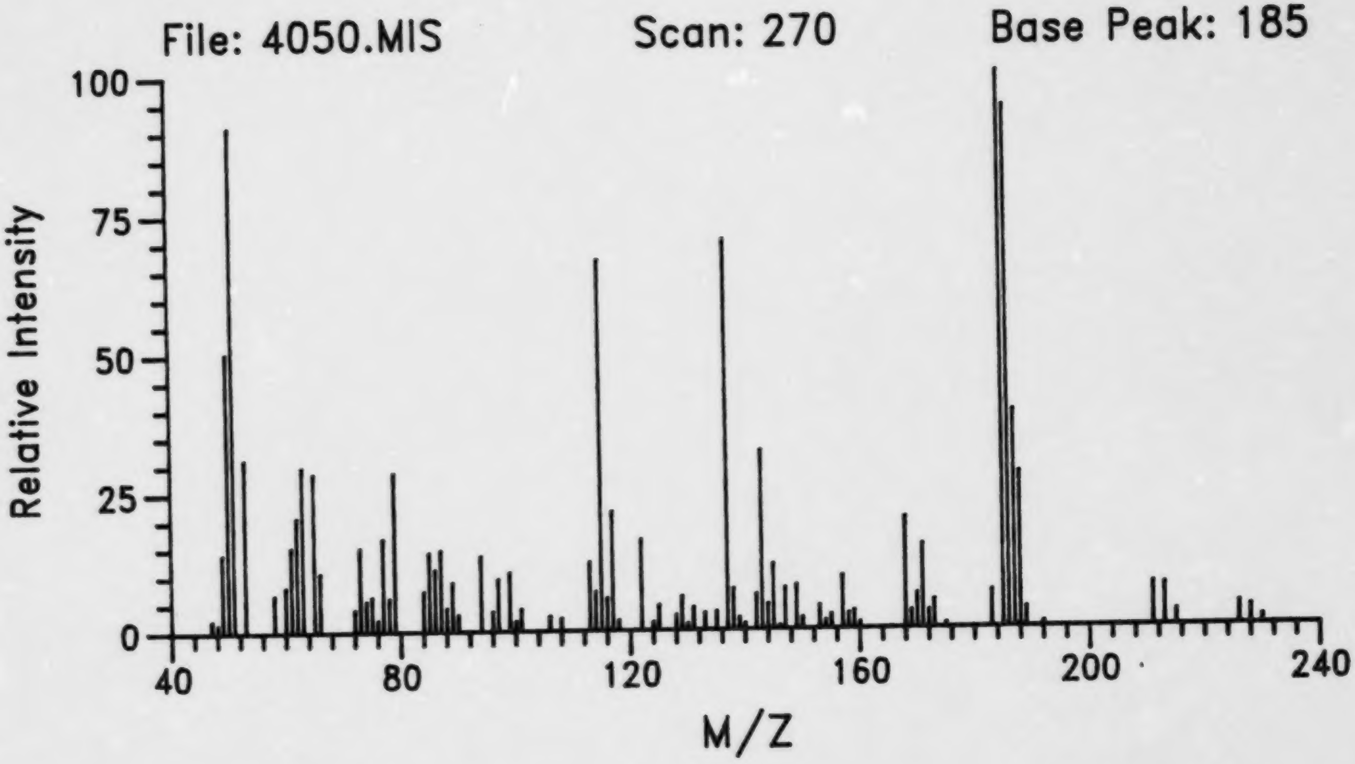


Figure A-17. Extract 4050 (Acid), Pulp and Paper Industry. Frequency of occurrence: 6.

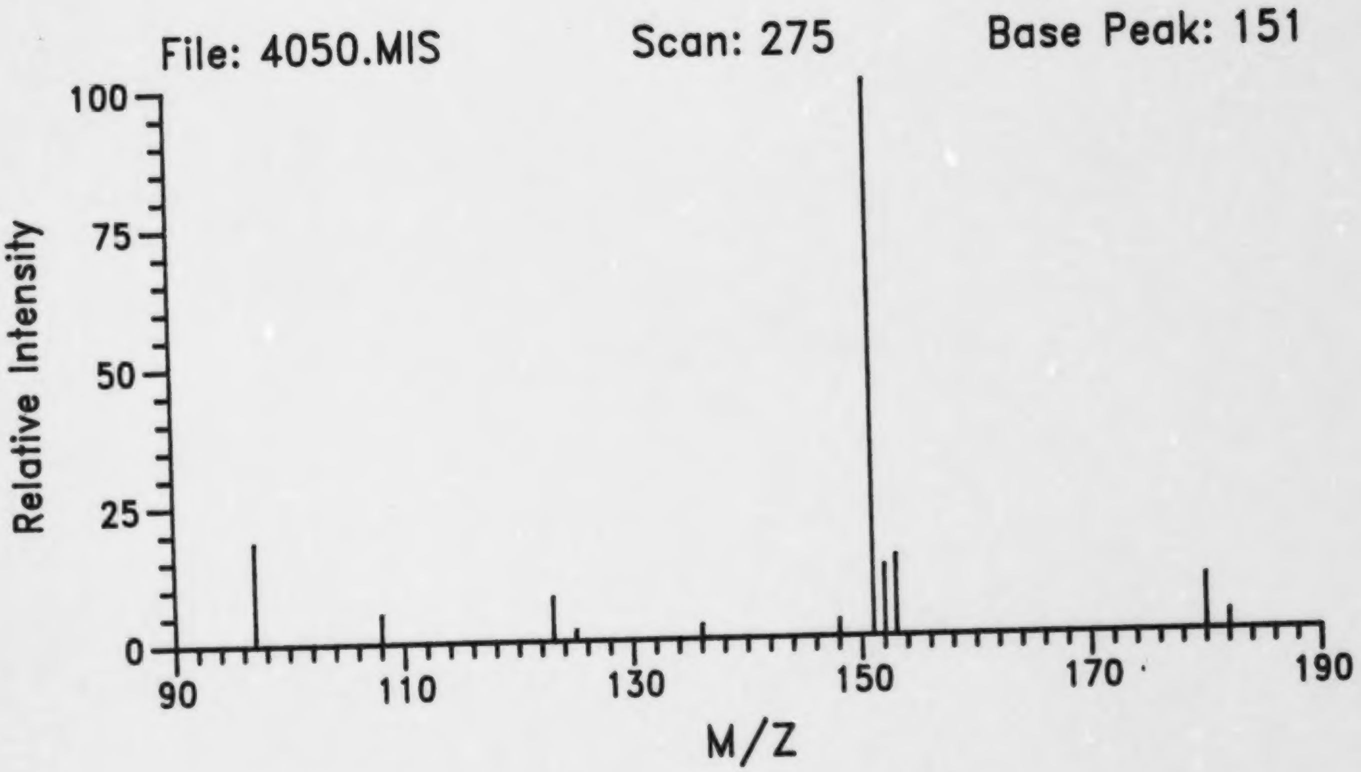


Figure A-18. Extract 4050 (Acid), Pulp and Paper Industry. Frequency of occurrence: 6.

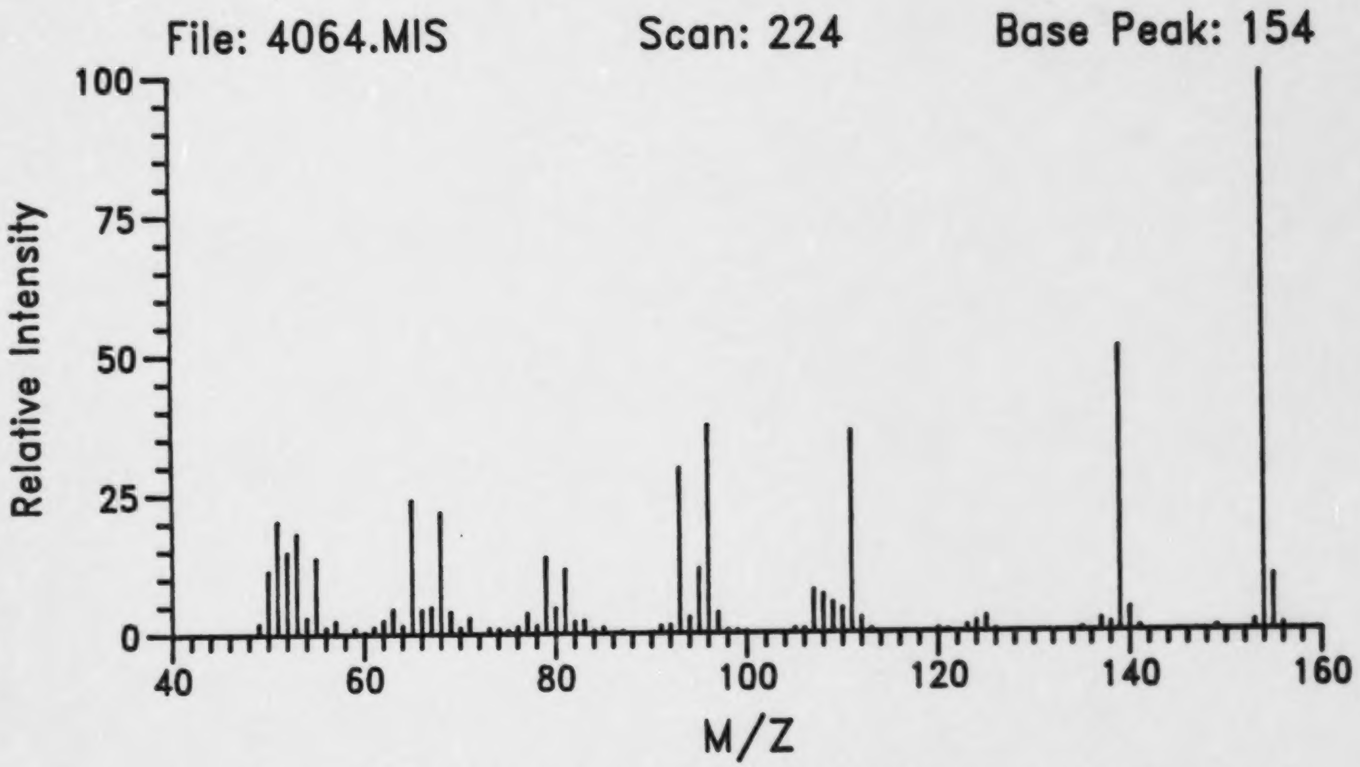


Figure A-19. Extract 4064 (acid), Pulp and Paper Industry. Frequency of occurrence: 5.

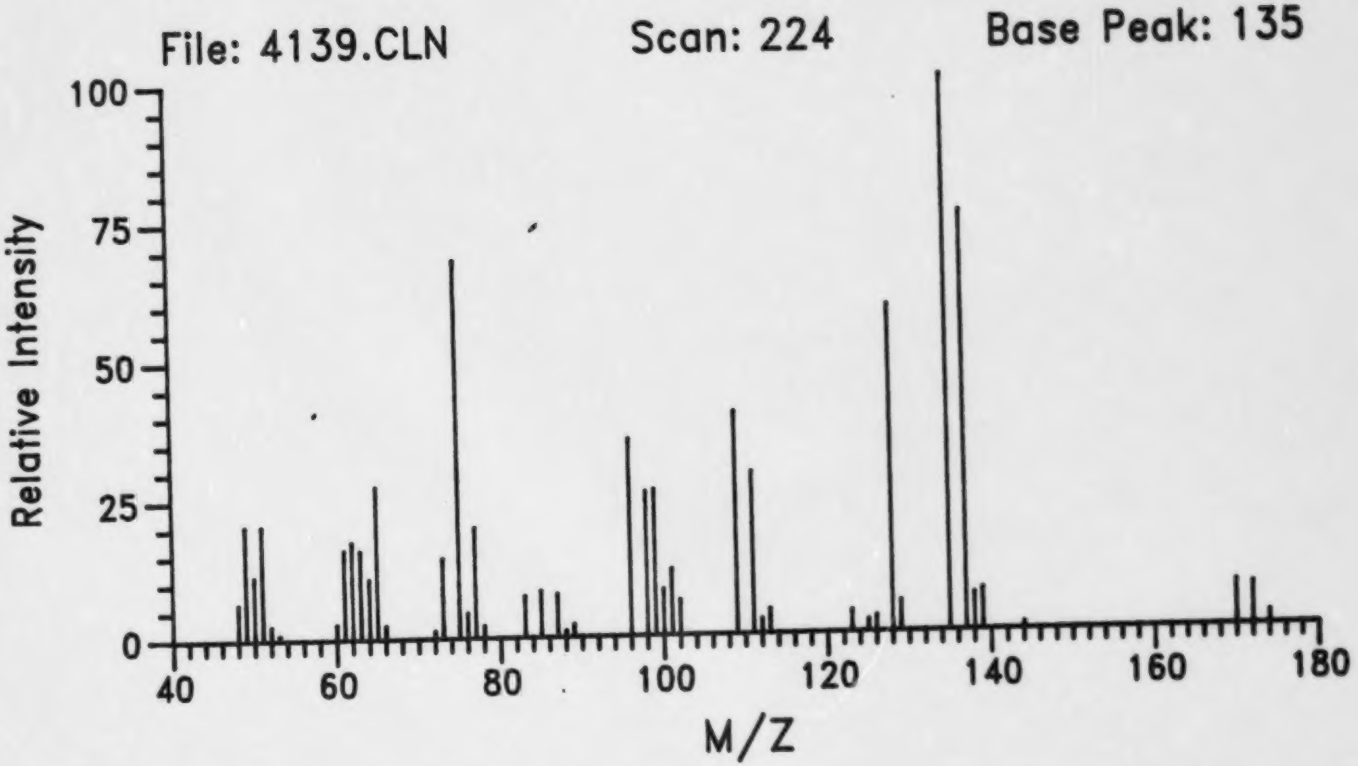


Figure A-20. Extract 4139 (Base/Neutral), Pesticides Manufacturing Industry. Frequency of occurrence: 5.

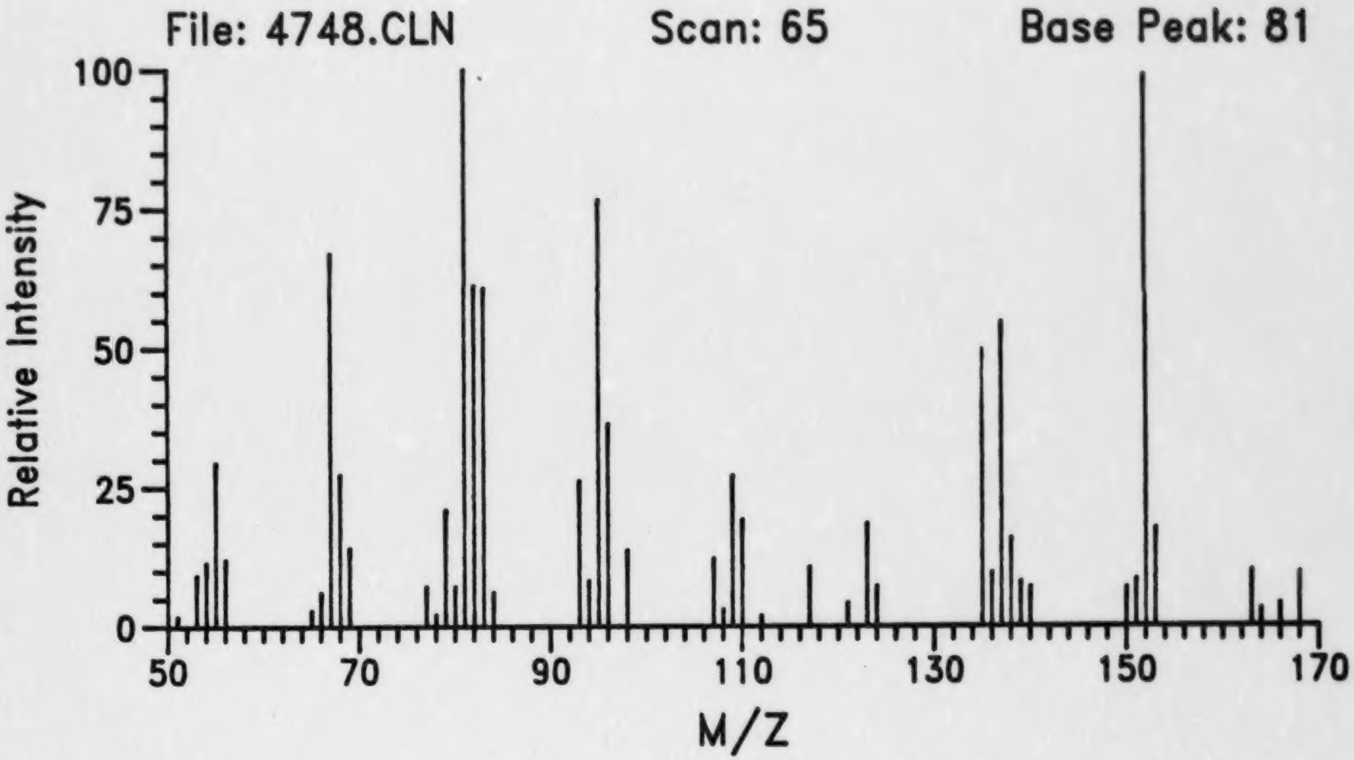


Figure A-21. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 6.

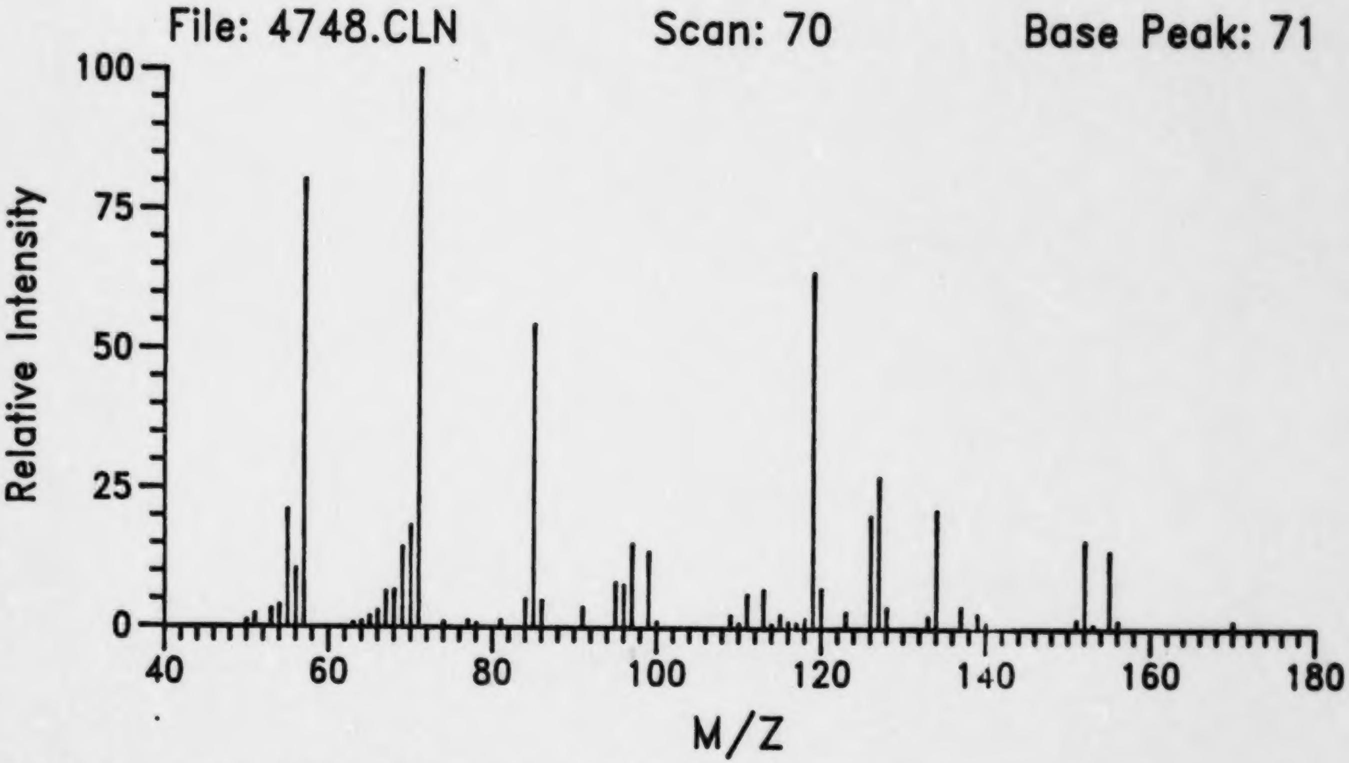


Figure A-22. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 6.

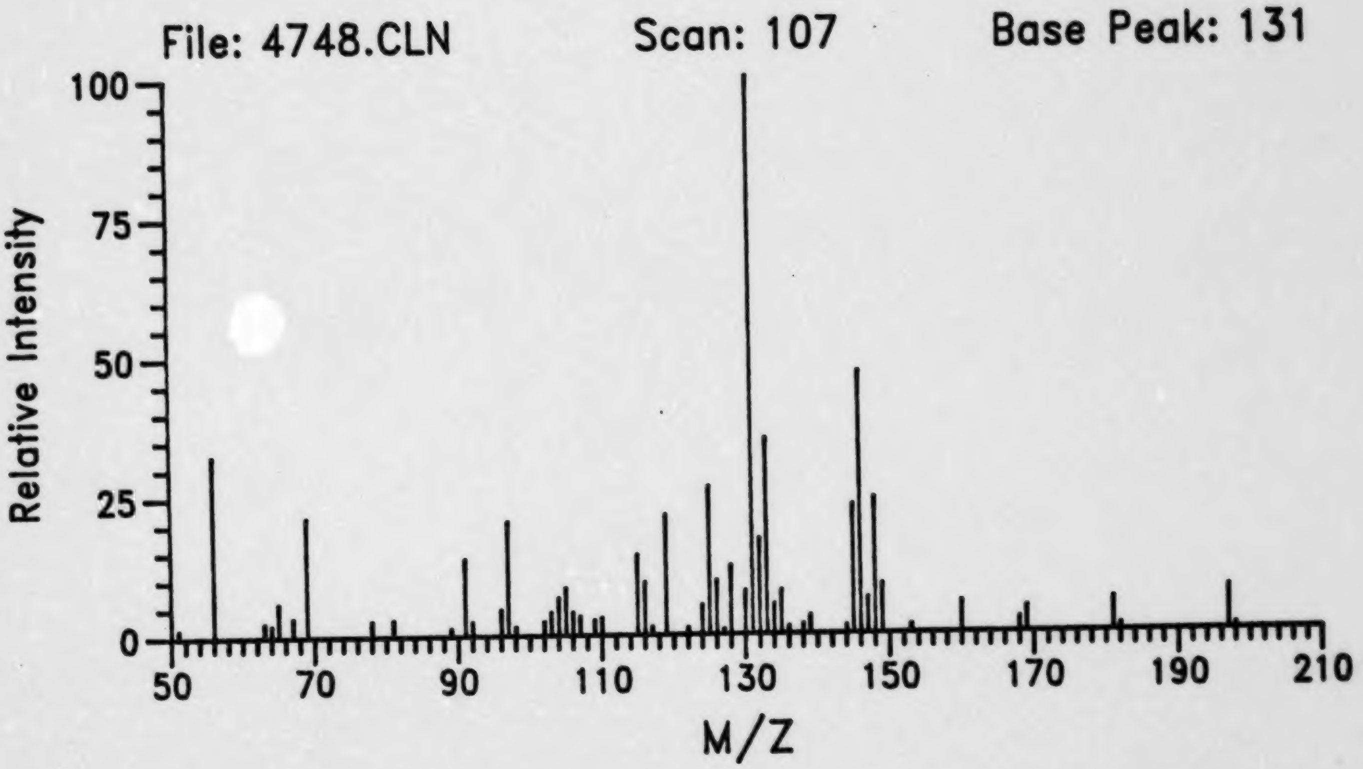


Figure A-23. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 5.

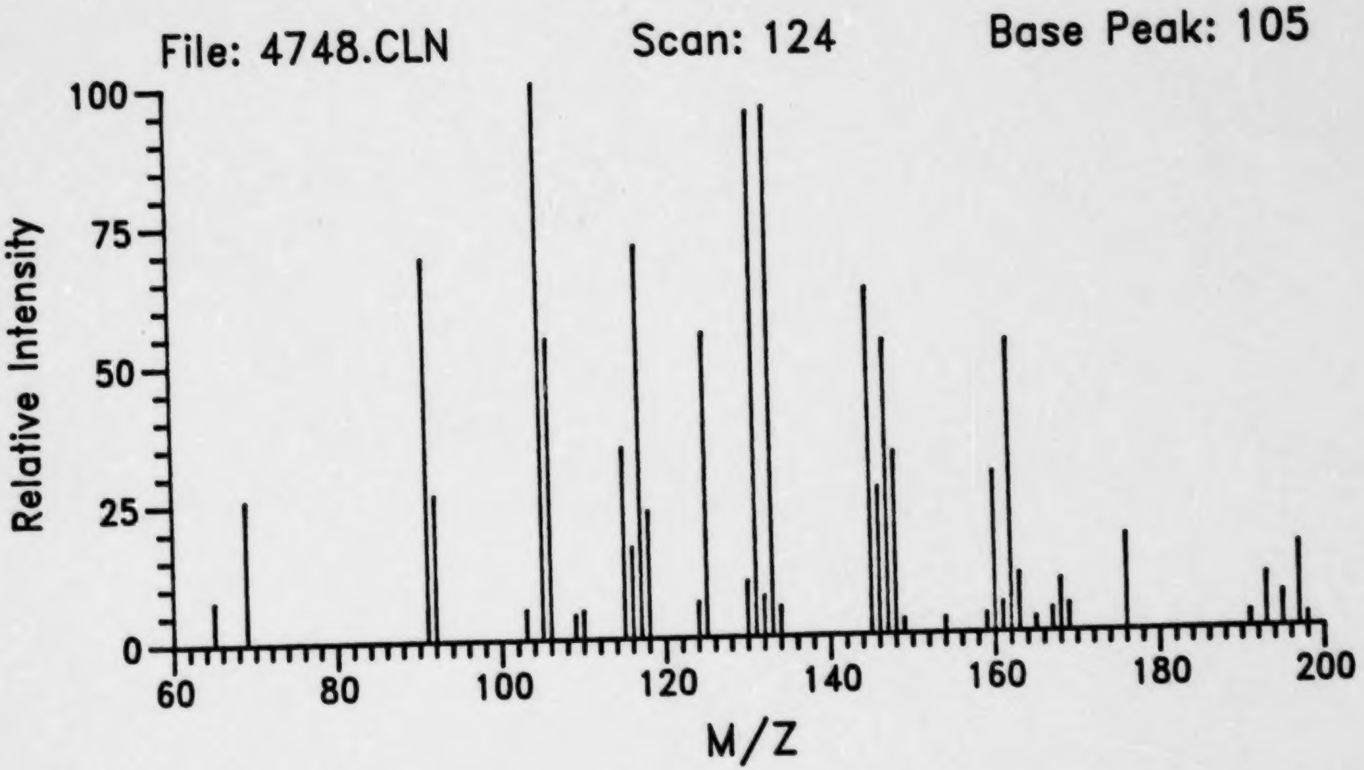


Figure A-24. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 5.

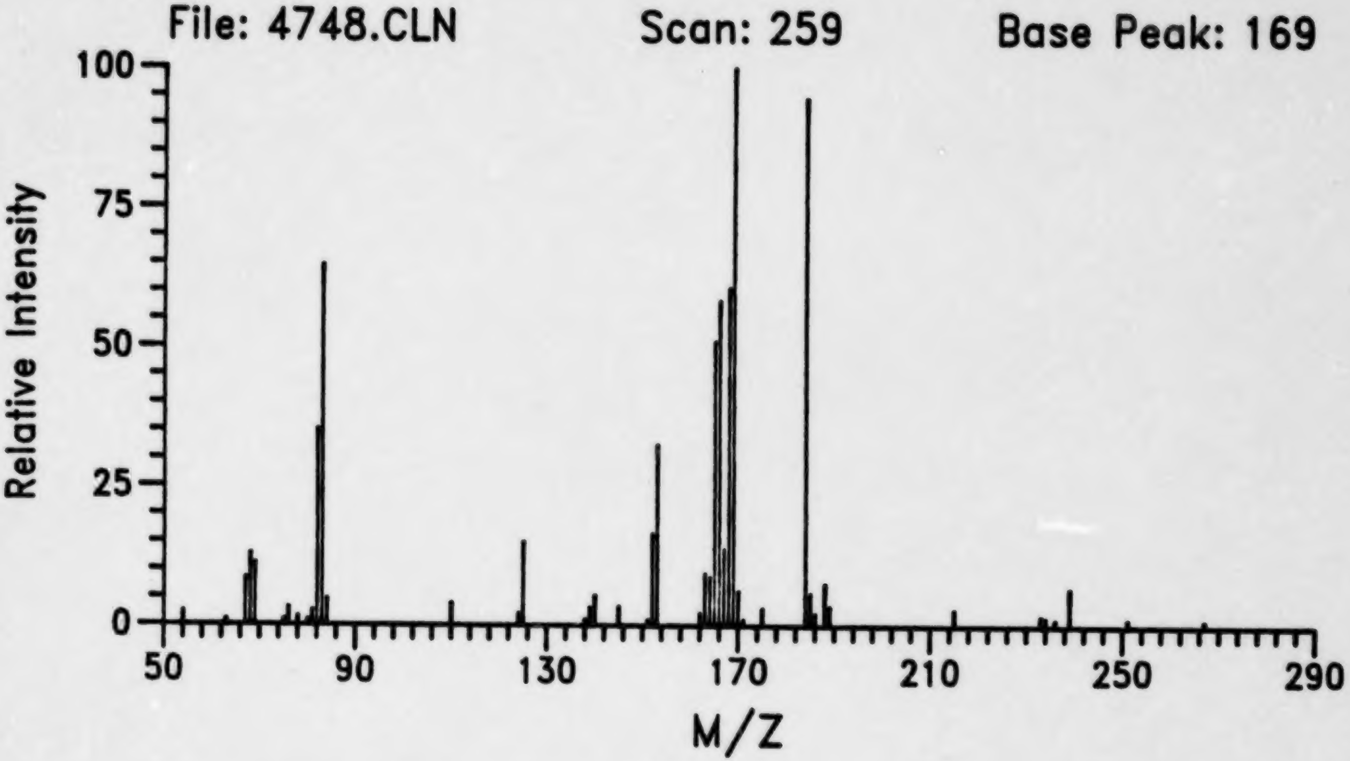


Figure A-25. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 9.

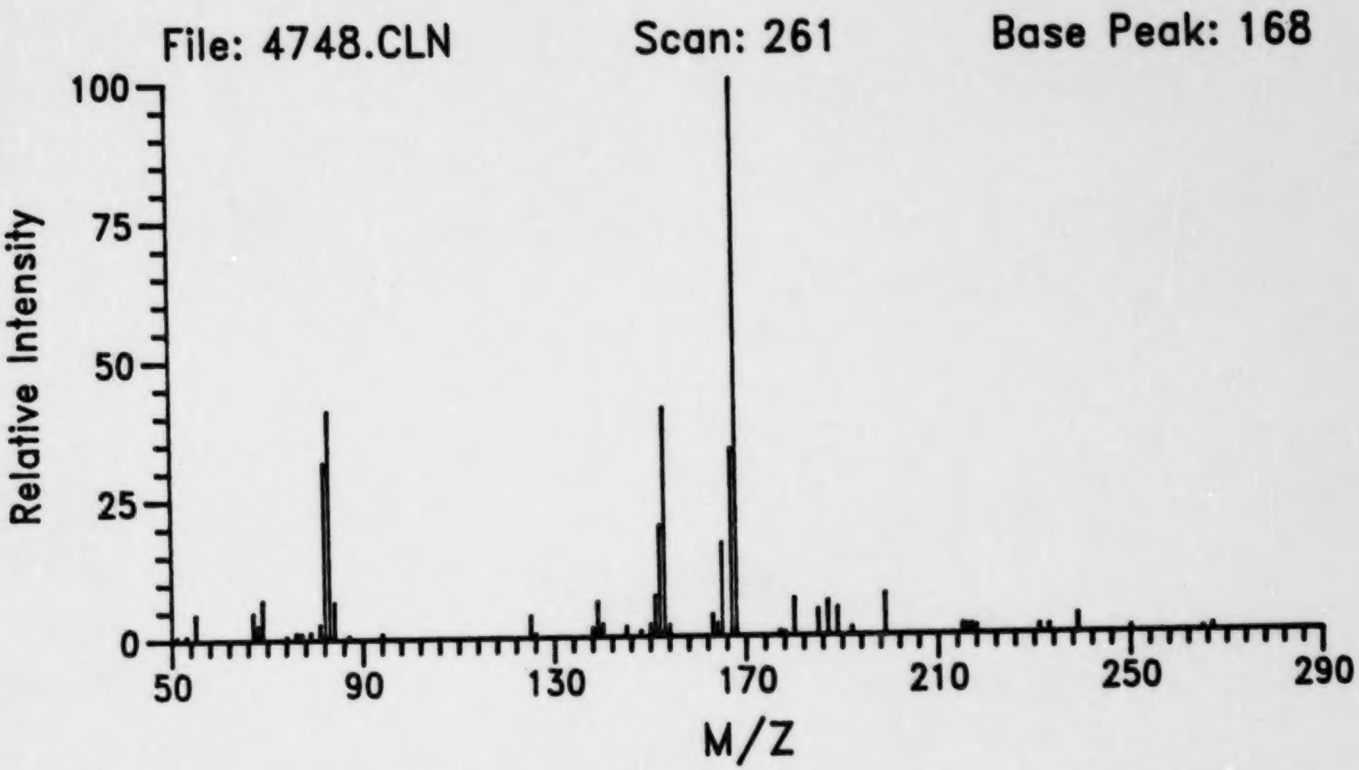


Figure A-26. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 22.

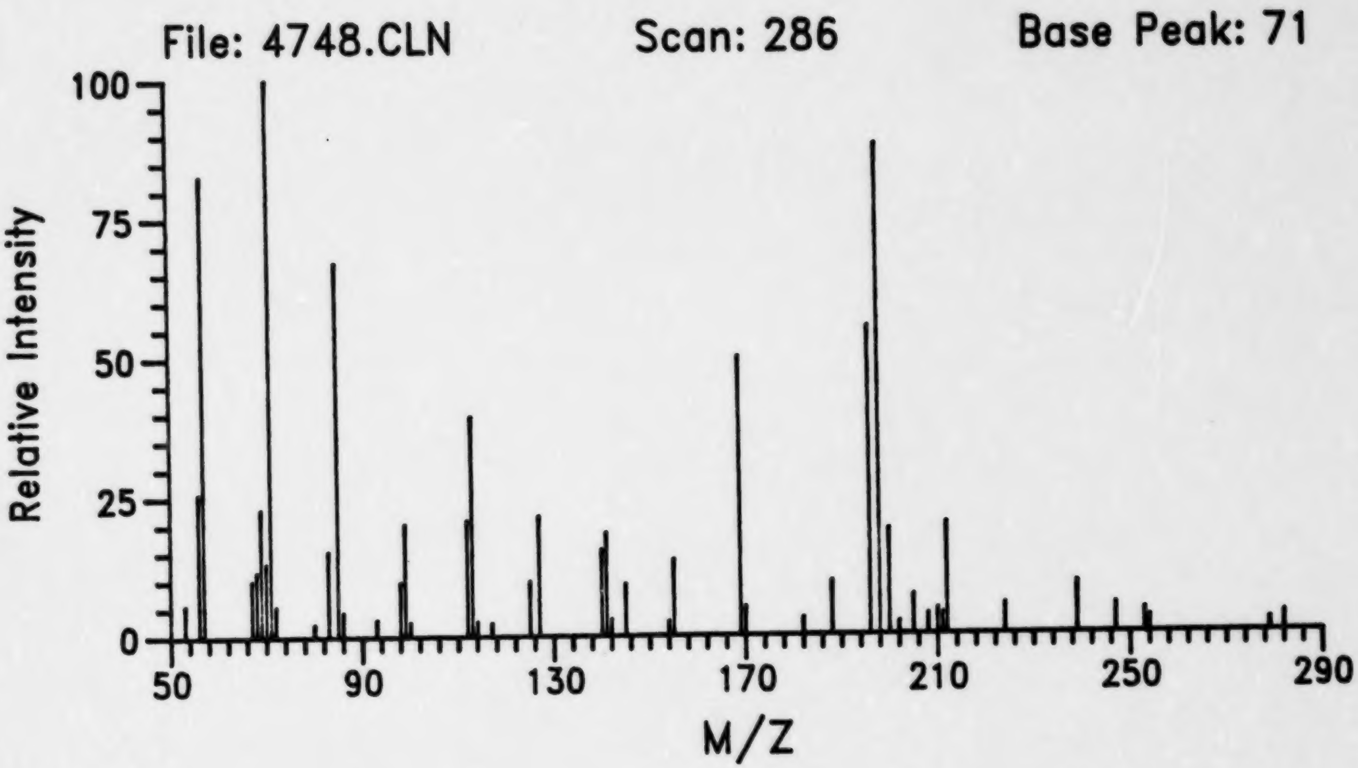


Figure A-27. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 6.

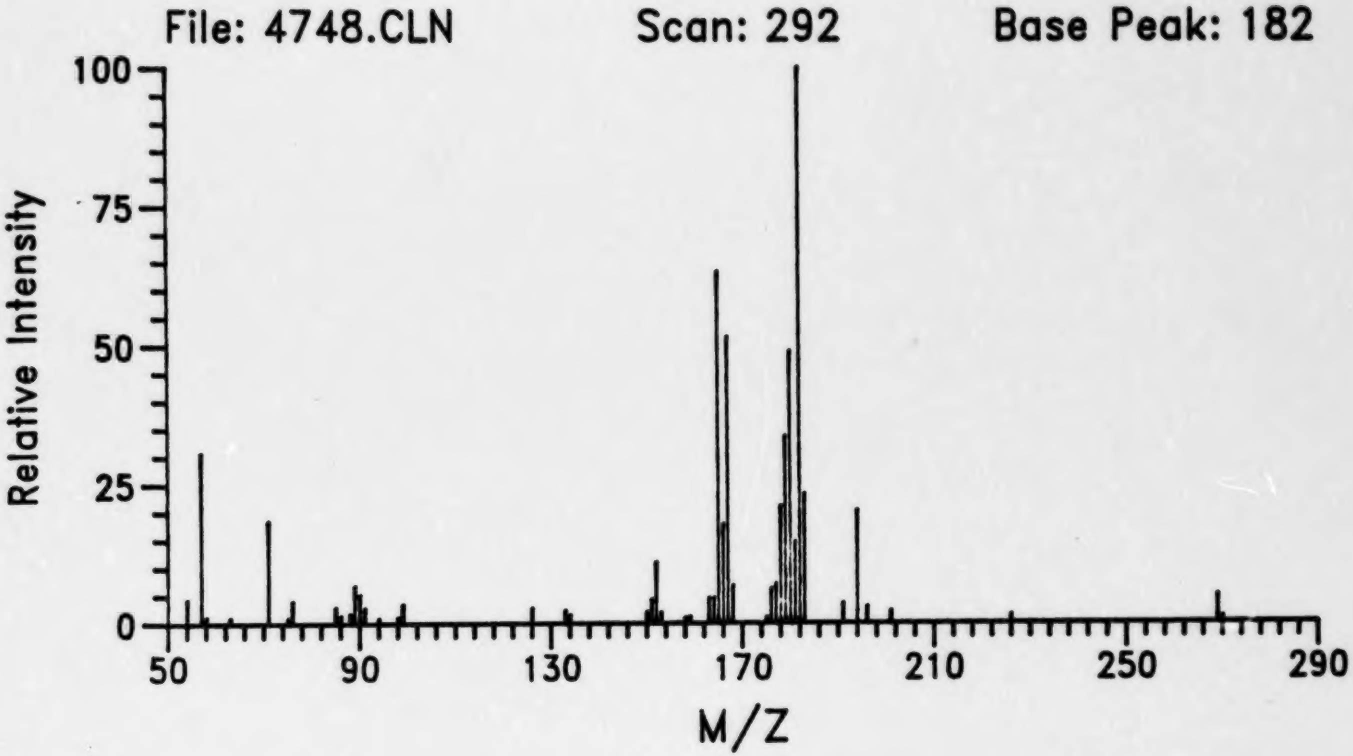


Figure A-28. Extract 4748 (base/neutral), electronics industry. Frequency of occurrence: 6.

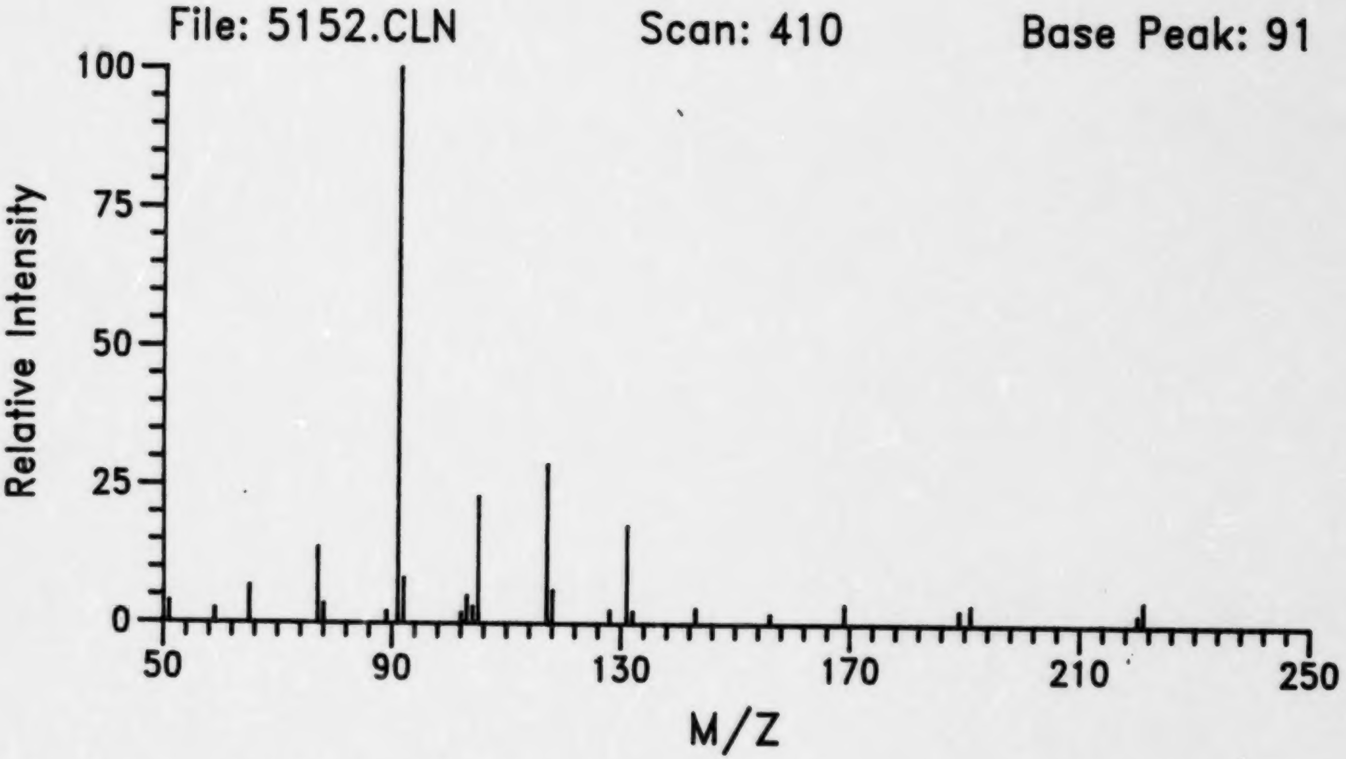


Figure A-29. Extract 5152 (Base/Neutral), Mechanical Products Industry. Frequency of occurrence: 15.

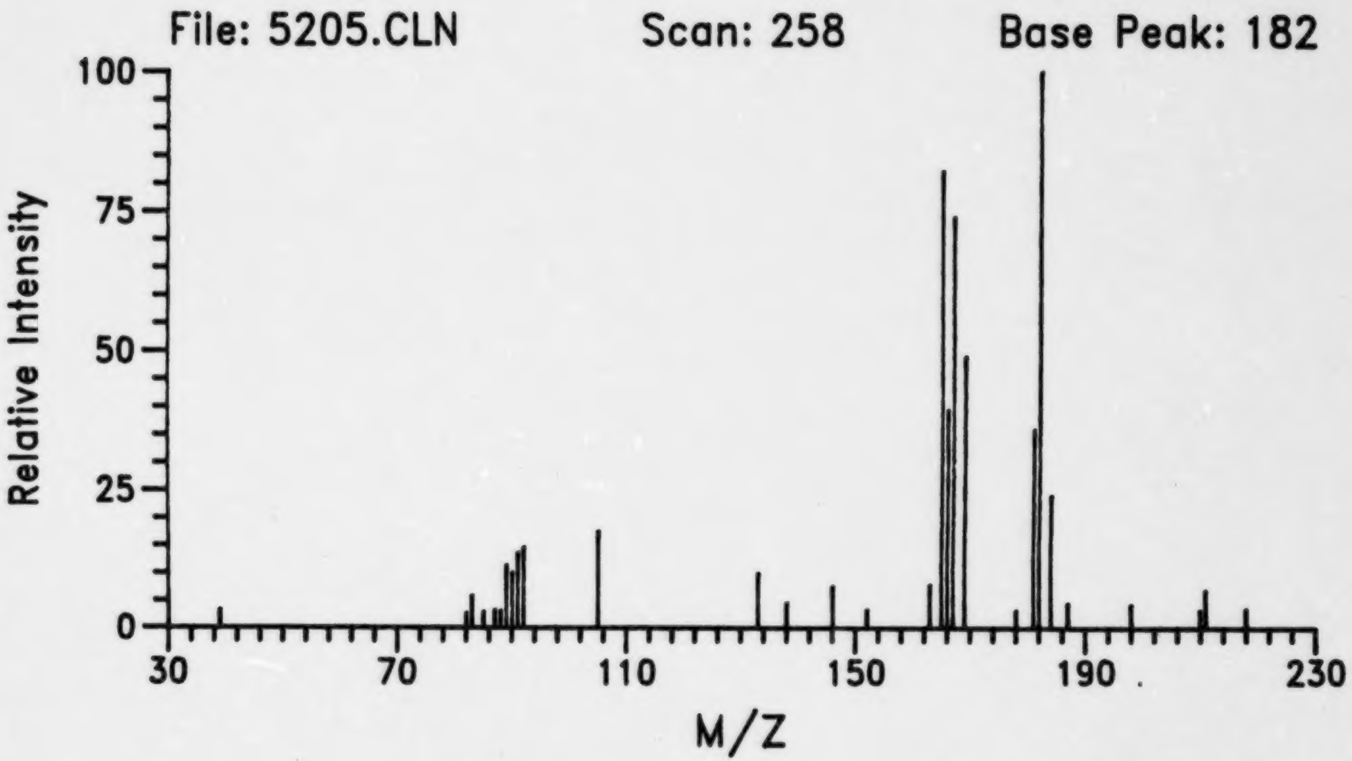


Figure A-30. Extract 5205 (Base/Neutral), Coal Mining Industry. Frequency of occurrence: 15.

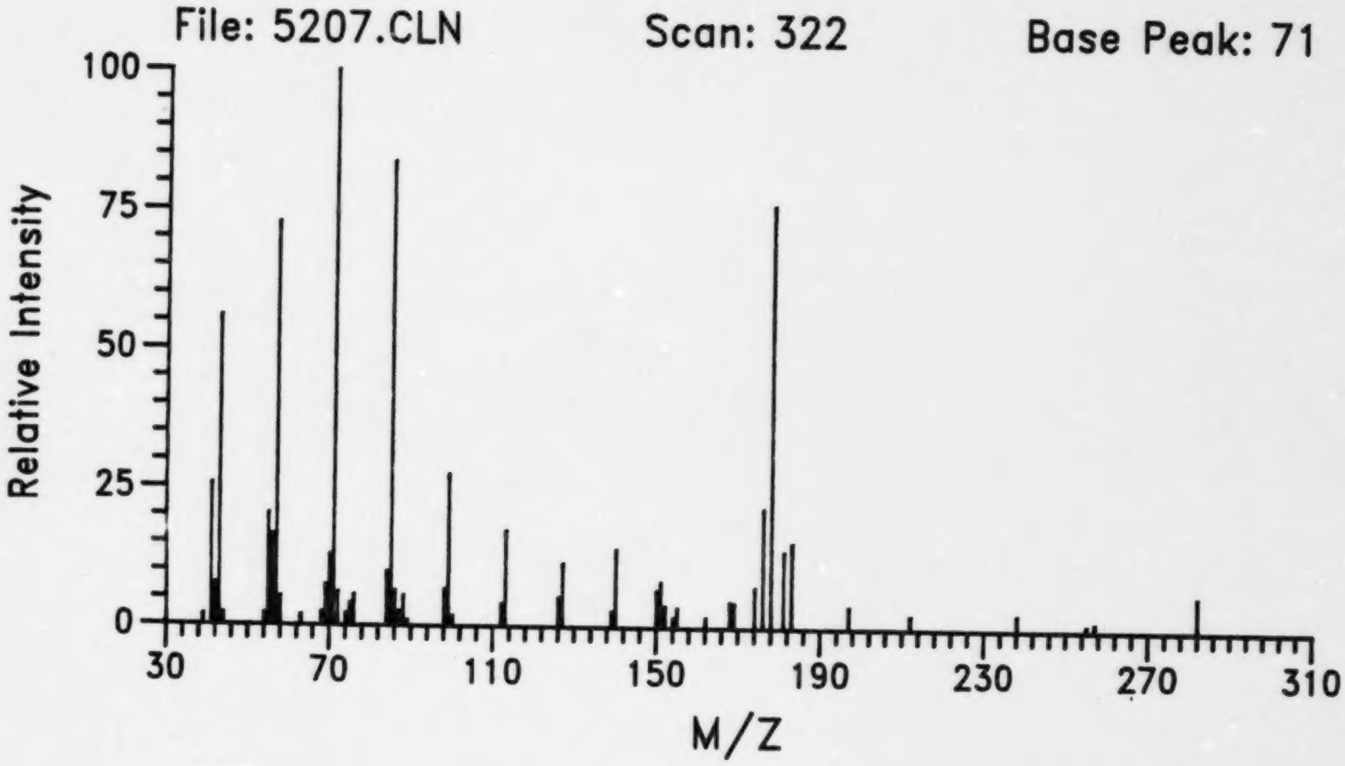


Figure A-31. Extract 5207 (Base/Neutral), Coal Mining Industry. Frequency of occurrence: 17.

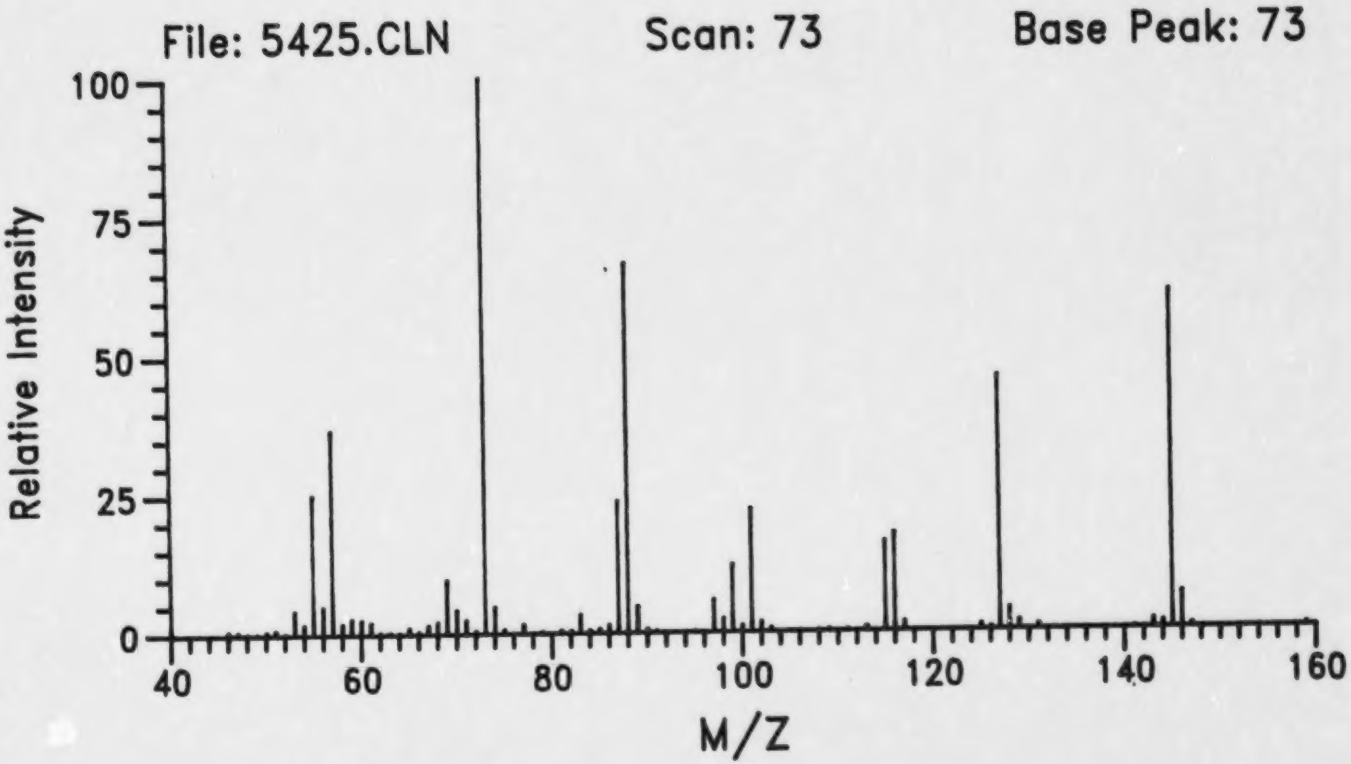


Figure A- 32. Extract 5425 (Acid), Mechanical Products Industry. Frequency of occurrence: 35.

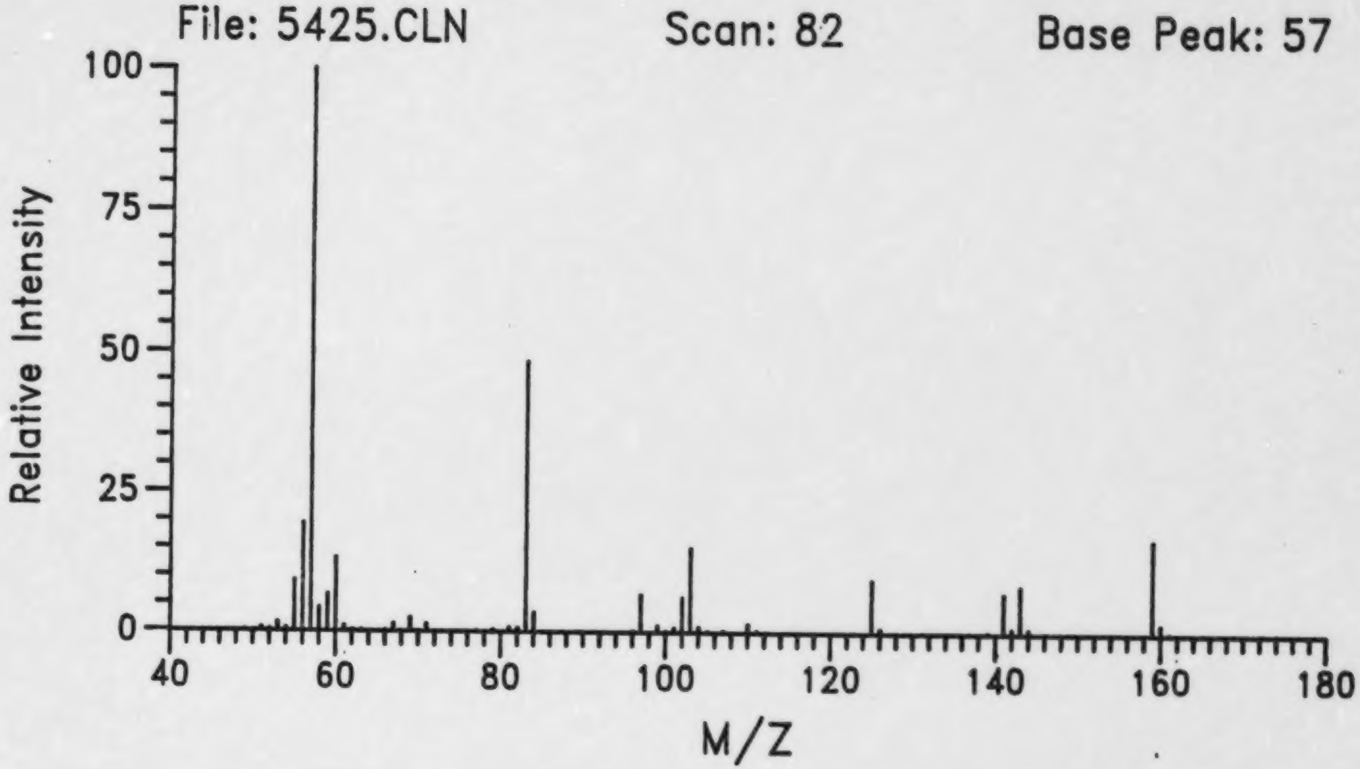


Figure A-33. Extract 5425 (Acid), Mechanical Products Industry. Frequency of occurrence: 22.

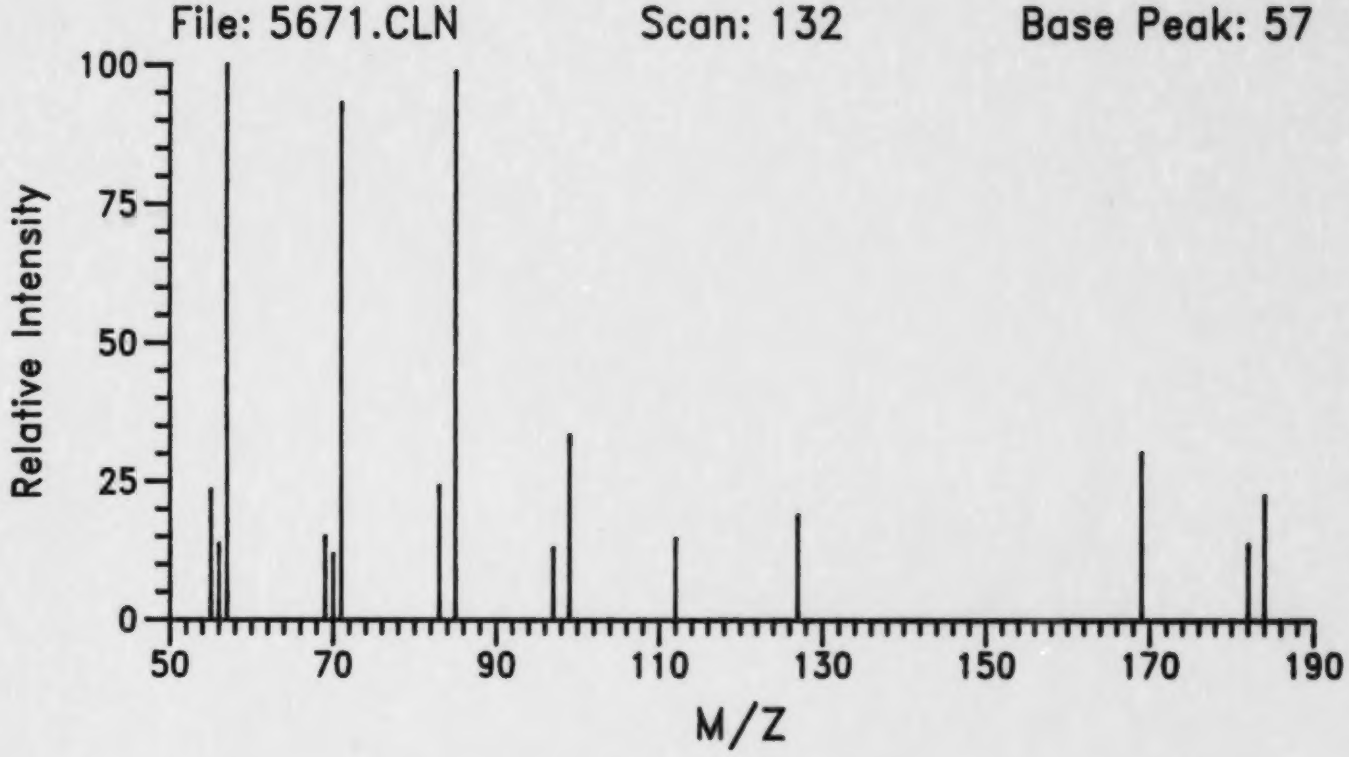


Figure A-34. Extract 5671 (Acid), Printing and Publishing Industry. Frequency of occurrence: 15.

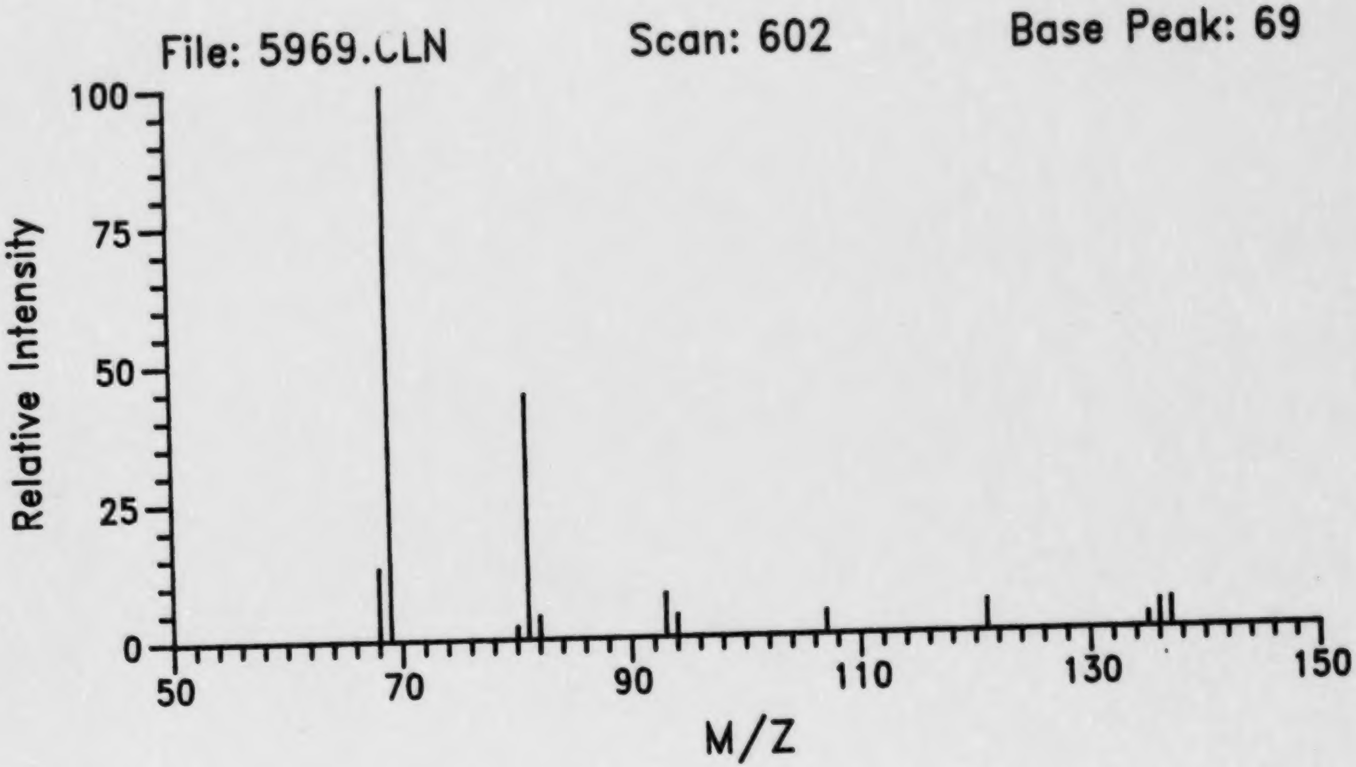


Figure A-35. Extract 5969 (Base/Neutral), Publicly Owned Treatment Works. Frequency of occurrence: 47.

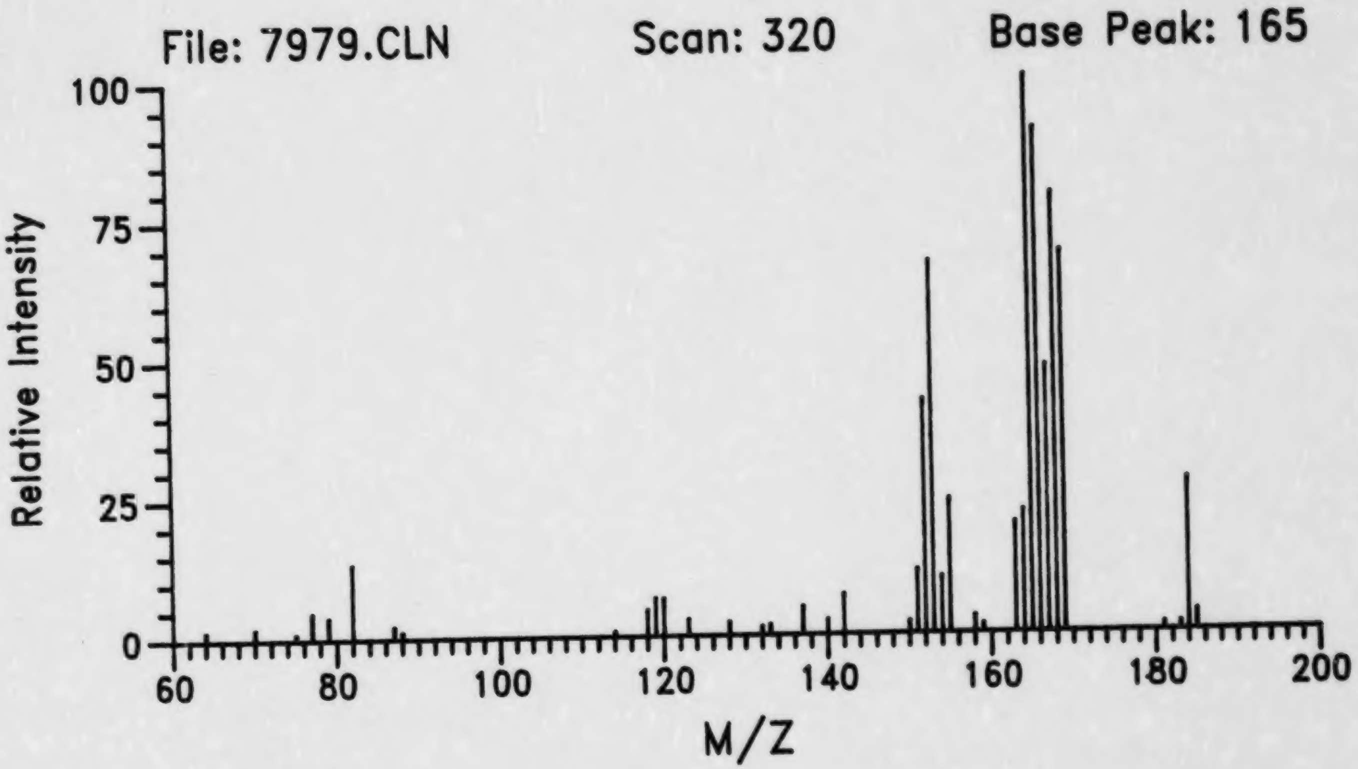


Figure A-36. Extract 7979 (Base/Neutral), Petroleum Refining Industry. Frequency of occurrence: 5.

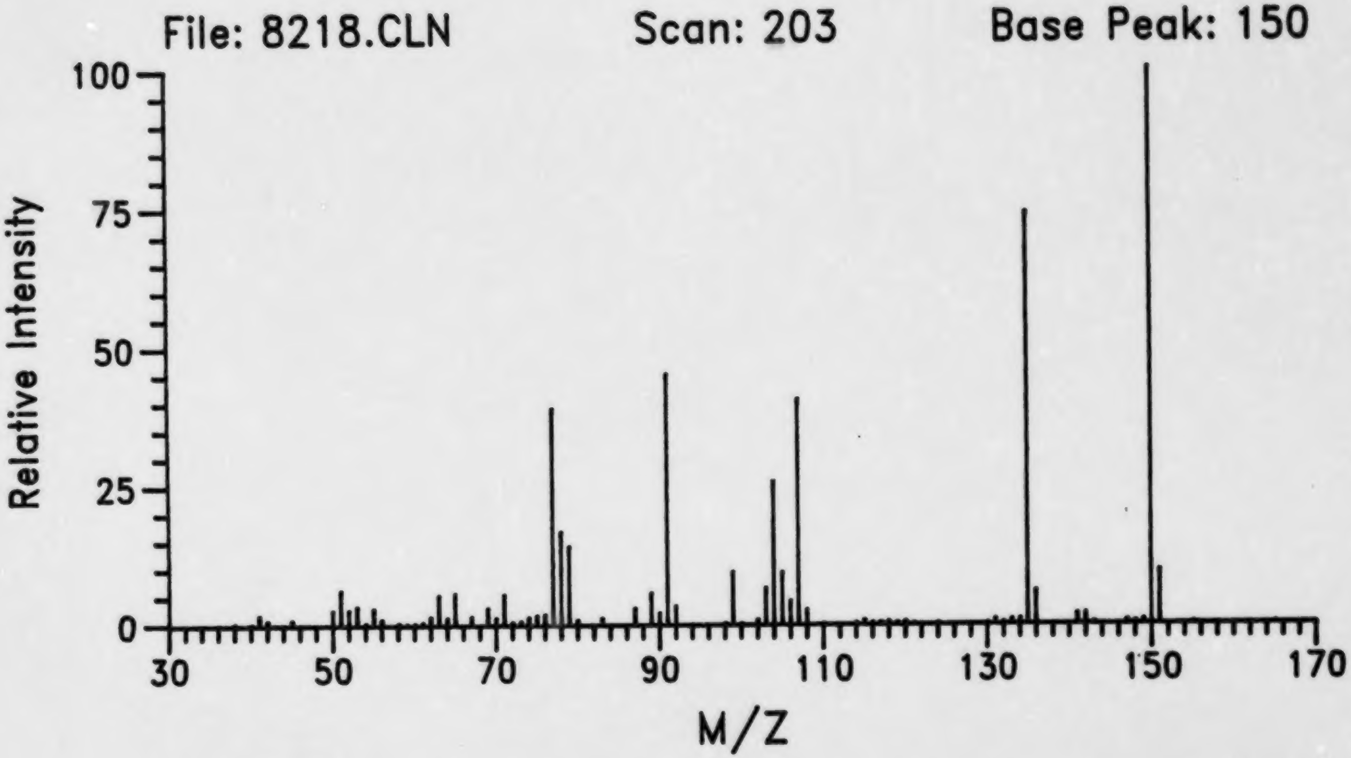


Figure A-37. Extract 8218 (Acid), Timber Products Industry. Frequency of occurrence: 9.

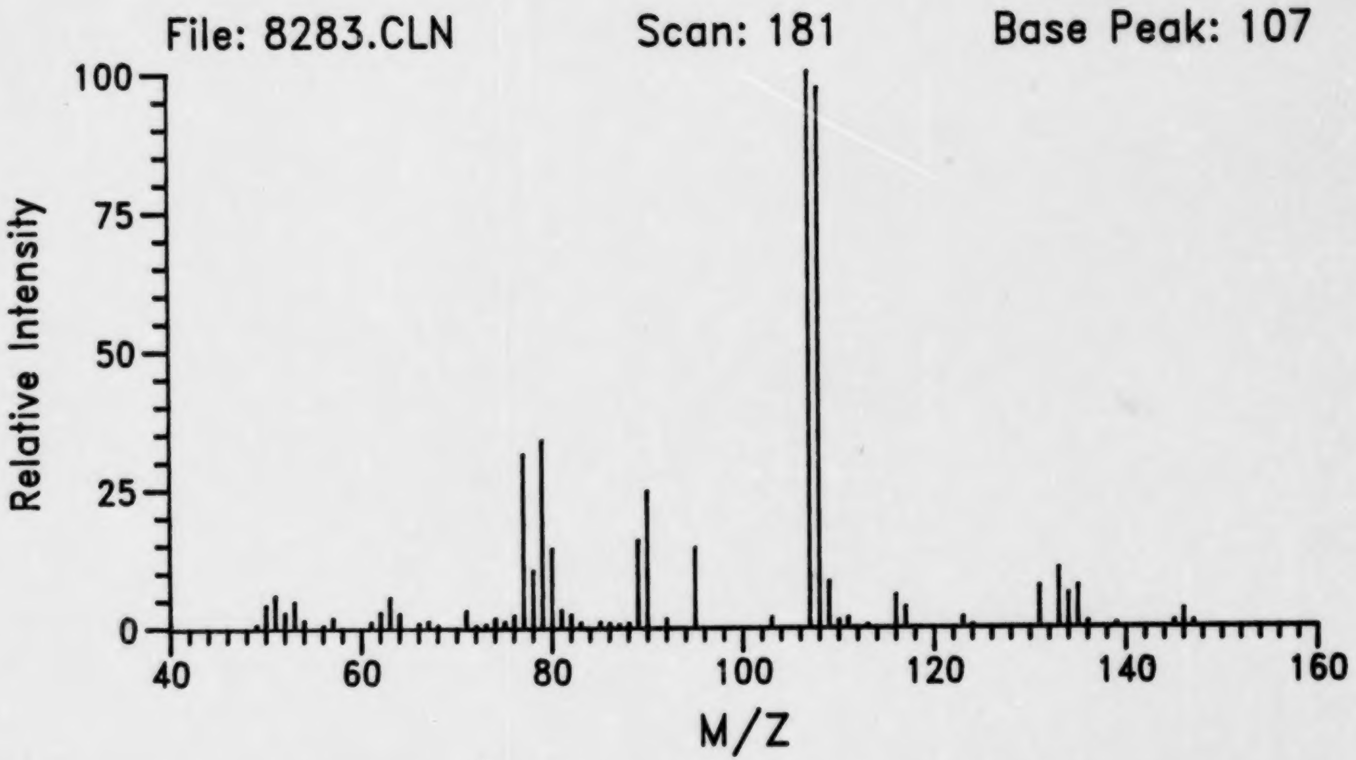


Figure A-38. Extract 8283 (Base/Neutral), Timber Products Industry. Frequency of occurrence: N/A.

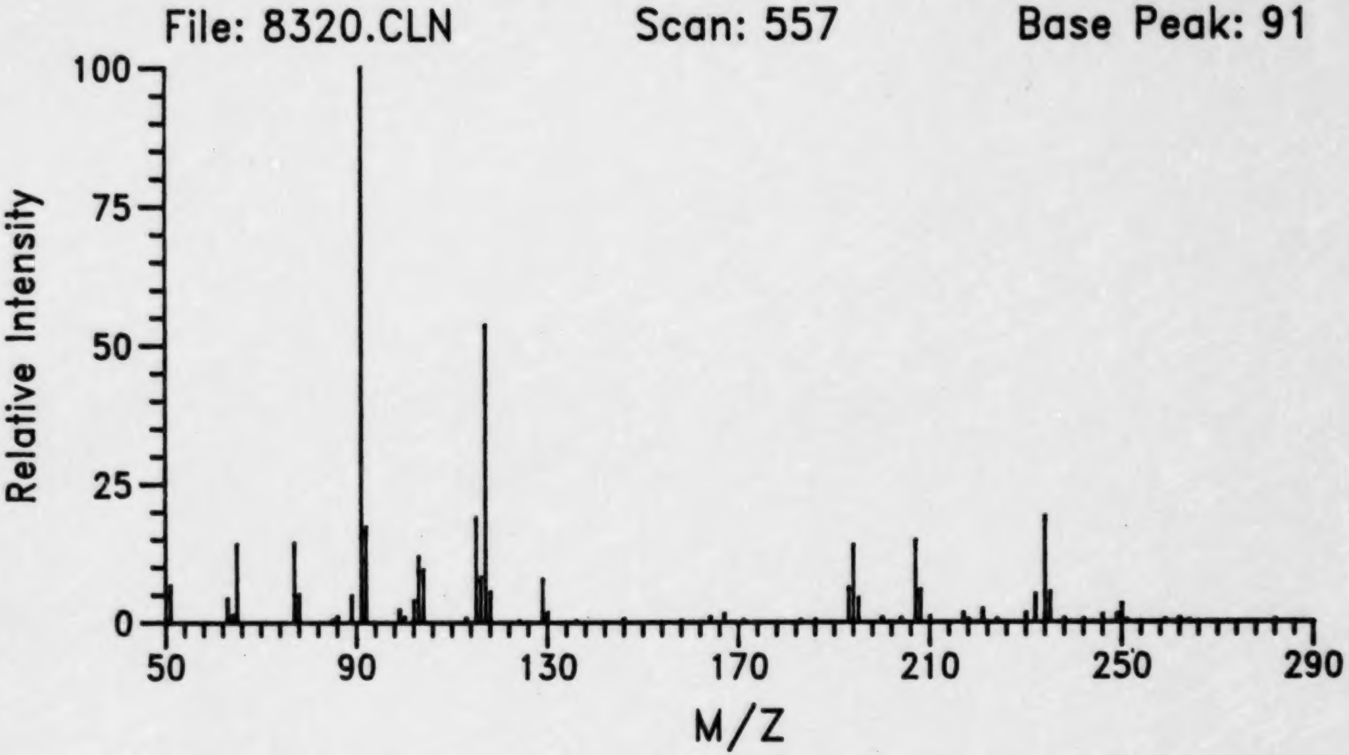


Figure A-39. Extract 8320 (Base/Neutral), Timber Products Industry. Frequency of occurrence: 58.

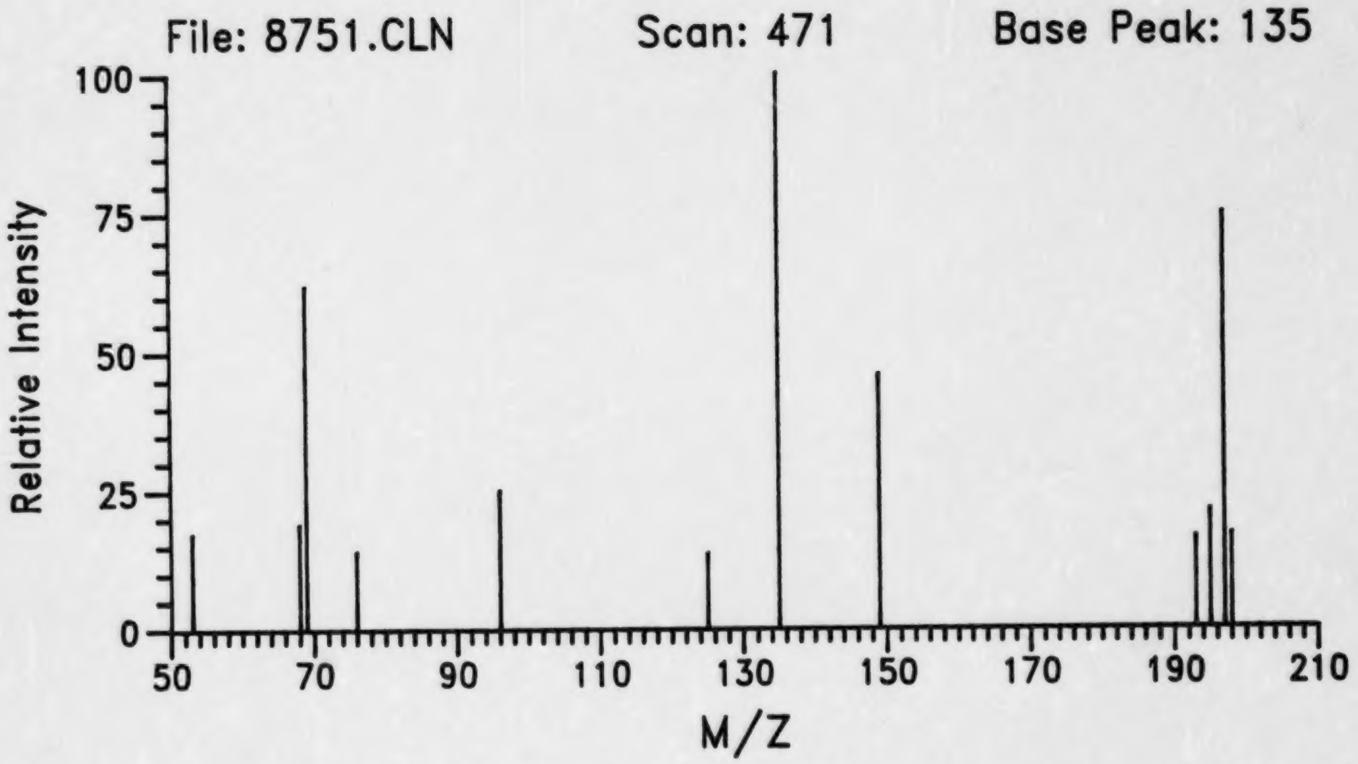


Figure A-40. Extract 8751 (Base/Neutral), Ore Mining Industry. Frequency of occurrence: 5.

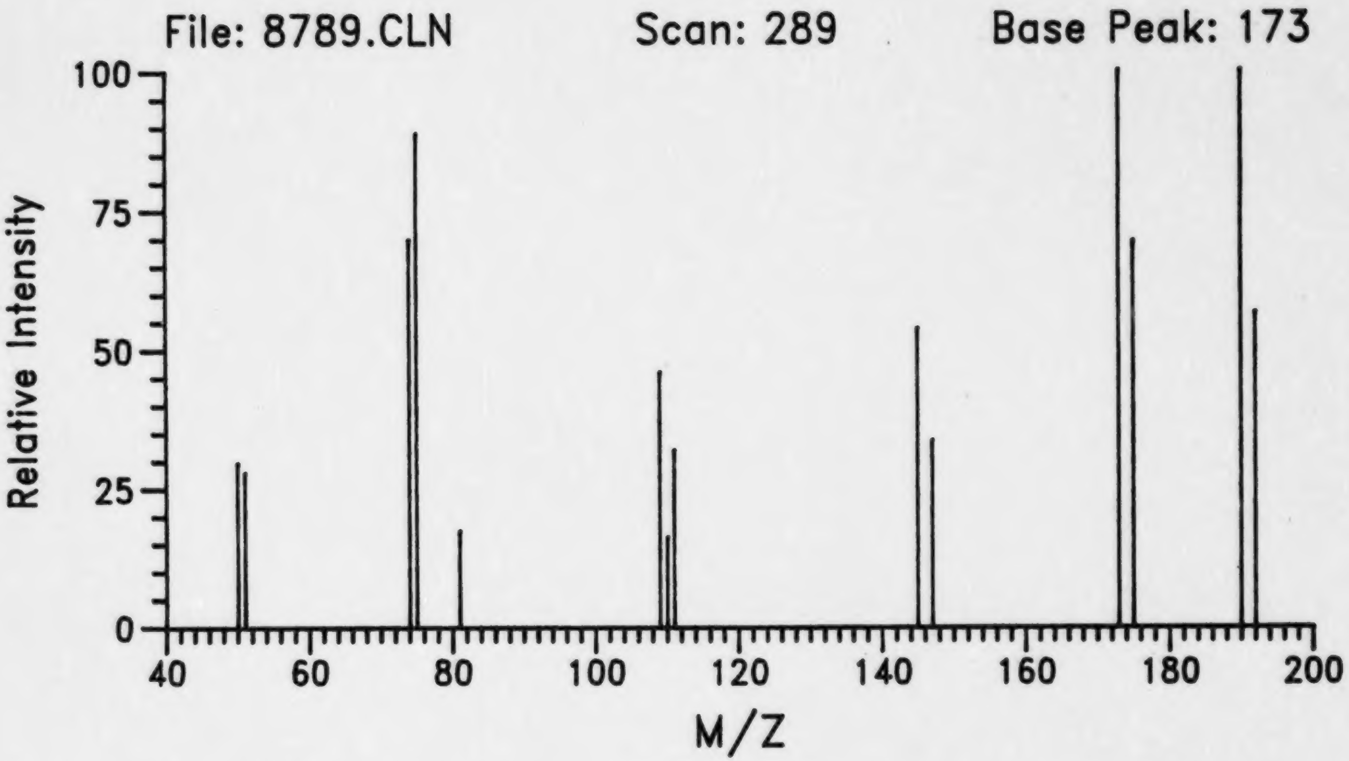


Figure A-41. Extract 8789 (Acid), Ore Mining Industry. Frequency of occurrence: N/A.

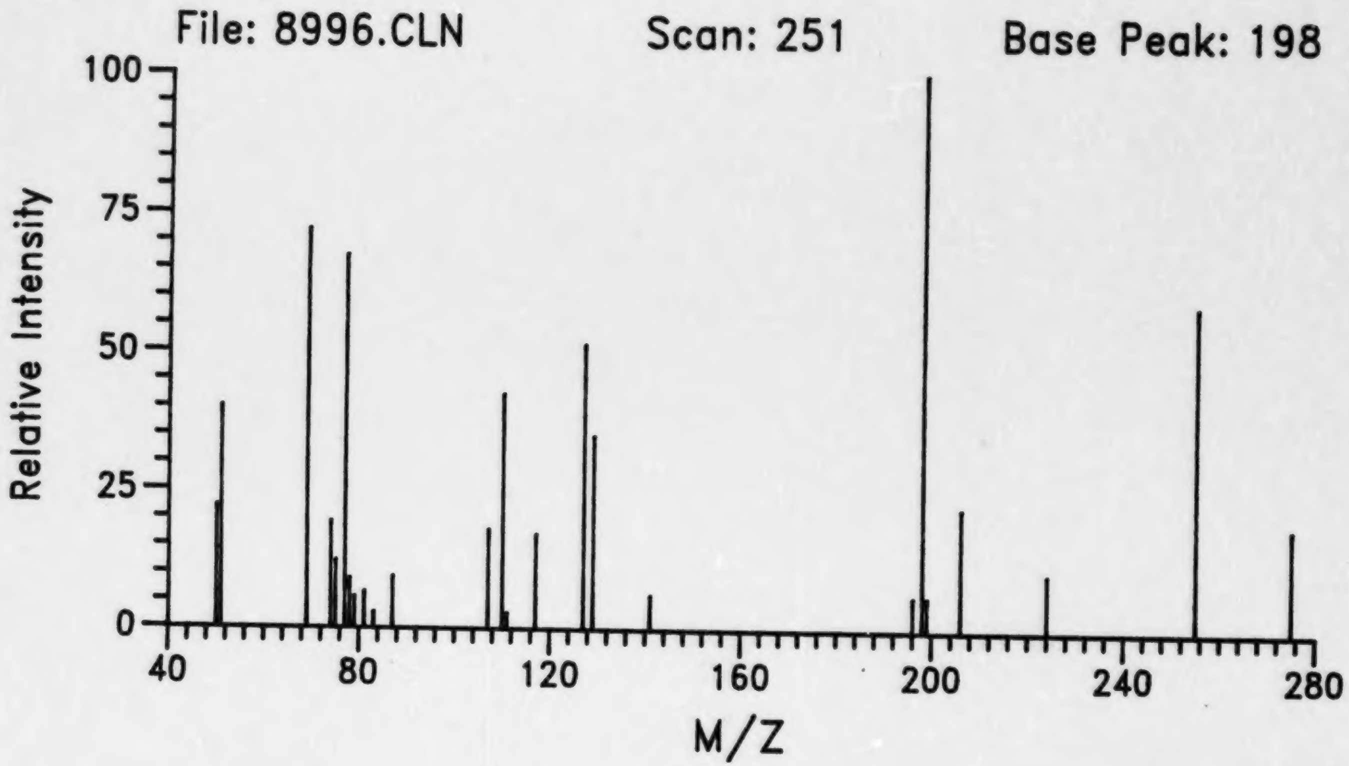


Figure A- 42. Extract 8996 (Acid), Publicly Owned Treatment Works. Frequency of occurrence: 37.

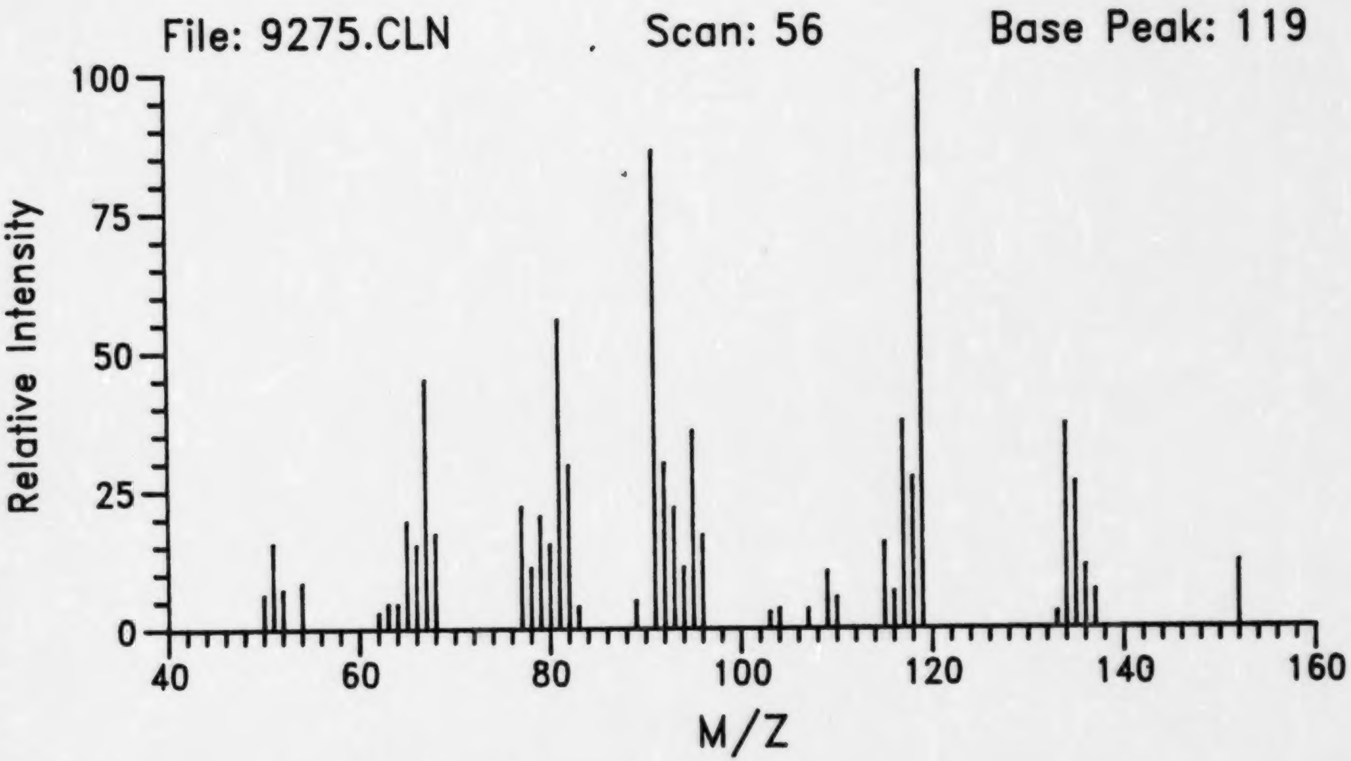


Figure A-43. Extract 9275 (Base/Neutral), Mechanical Products Industry. Frequency of occurrence: 6.

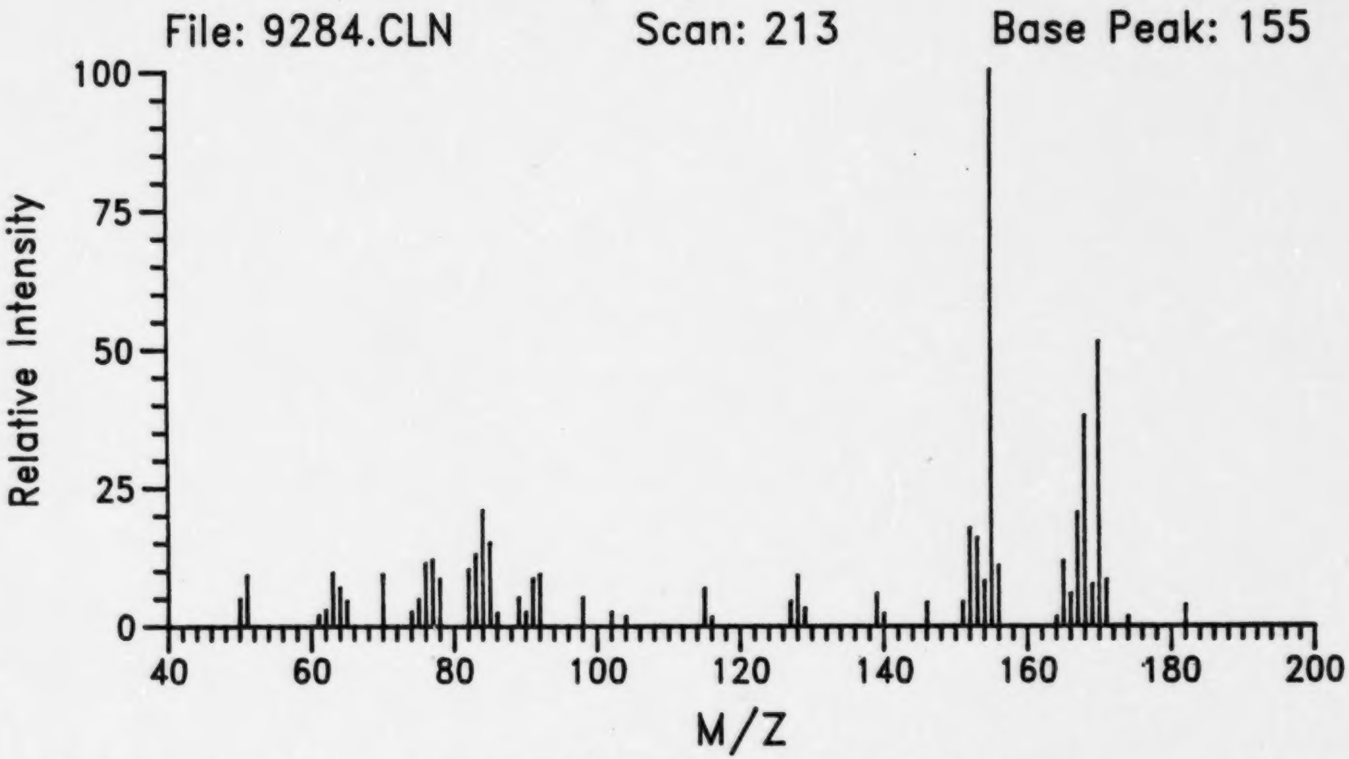


Figure A- 44. Extract 9284 (Base/Neutral), Mechanical Products Industry. Frequency of occurrence: 10.

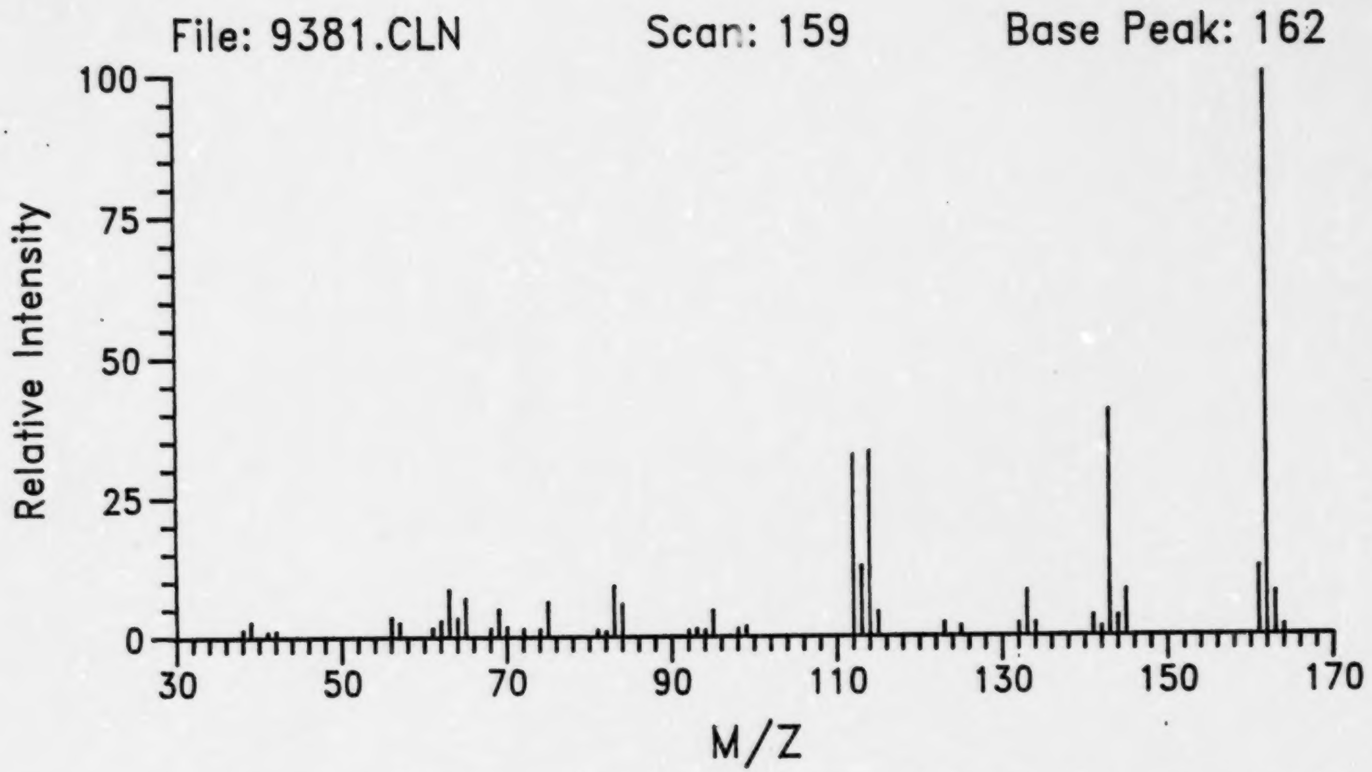


Figure A-45. Extract 9381 (Base/Neutral), Soaps and Detergents Industry. Frequency of occurrence: N/A.

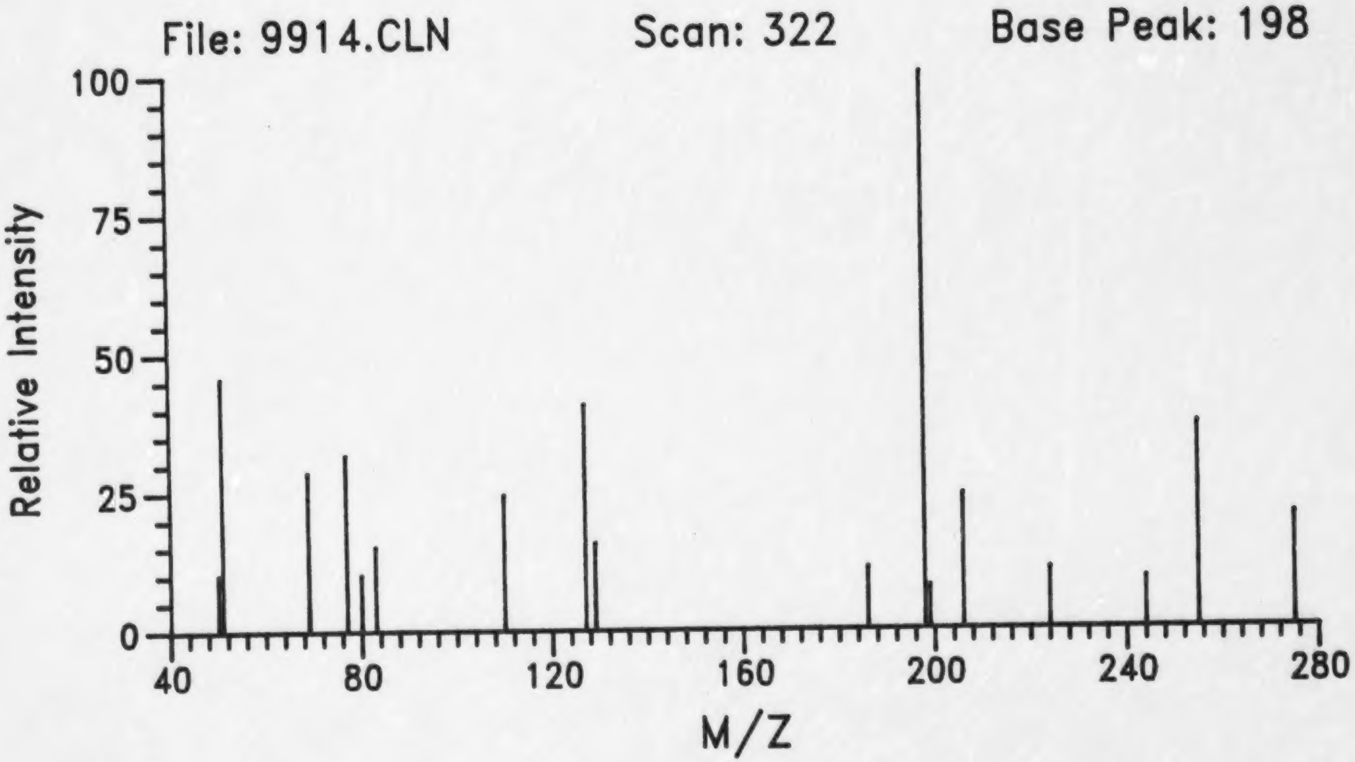


Figure A-46. Extract 9914 (Base/Neutral), Inorganic Chemicals Industry. Frequency of occurrence: 12.

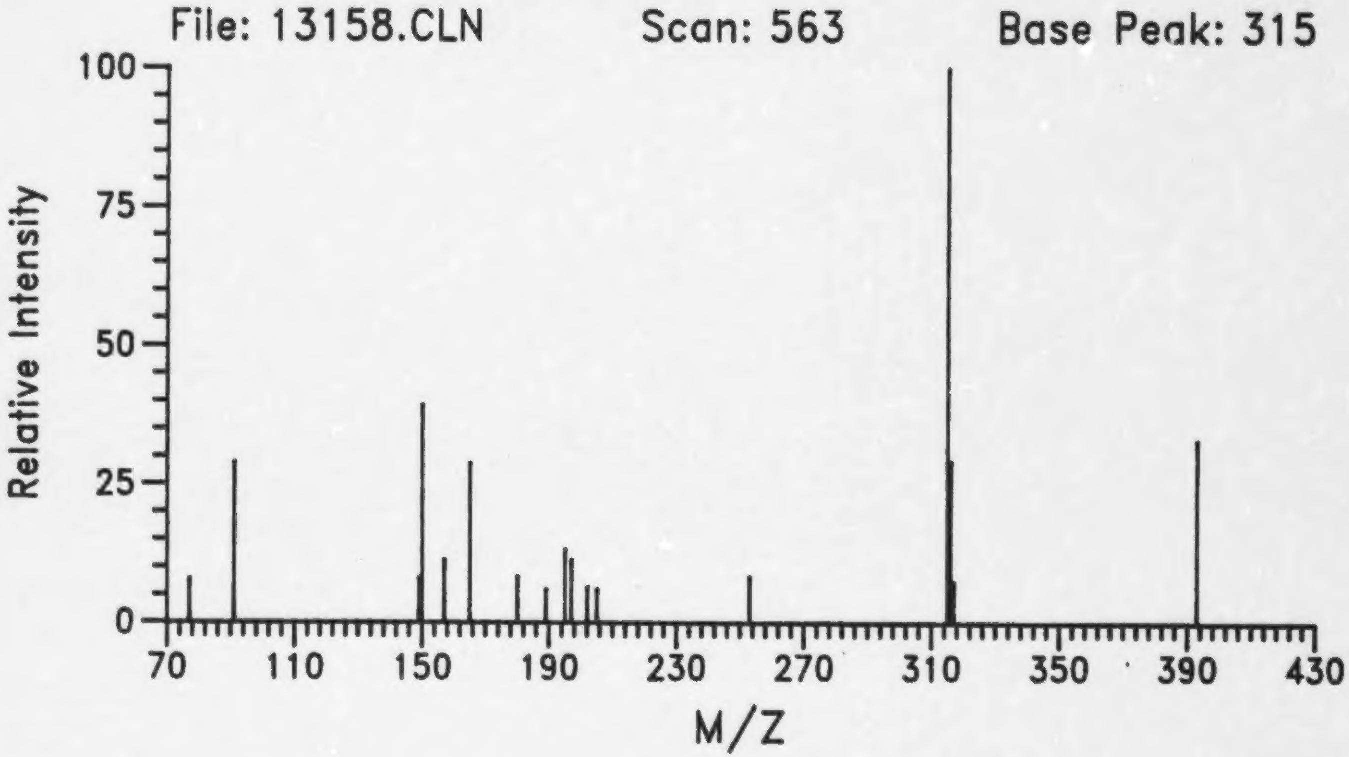


Figure A-47. Extract 13158 (Base/Neutral), Publicly Owned Treatment Works. Frequency of occurrence: N/A.

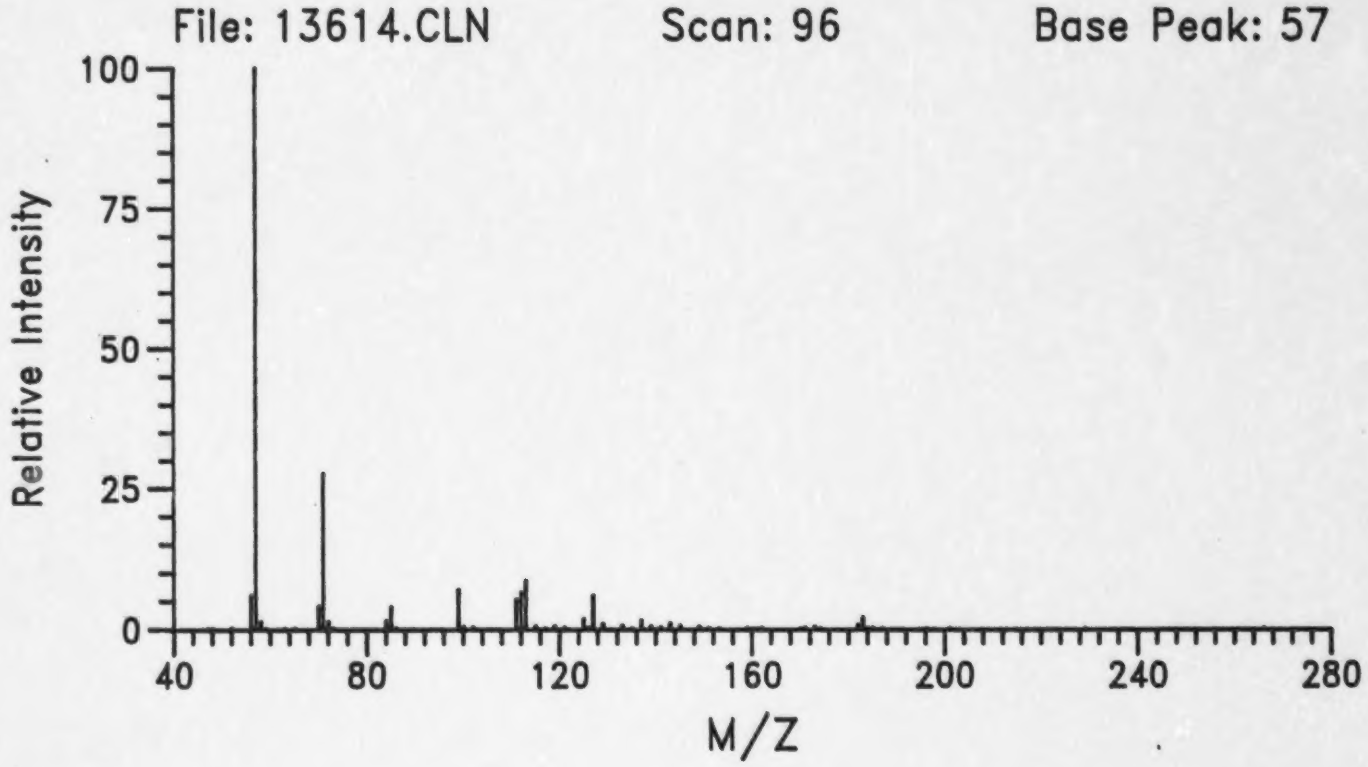


Figure A-48. Extract 13614 (Acid), Iron and Steel Manufacturing Industry. Frequency of occurrence: 14.

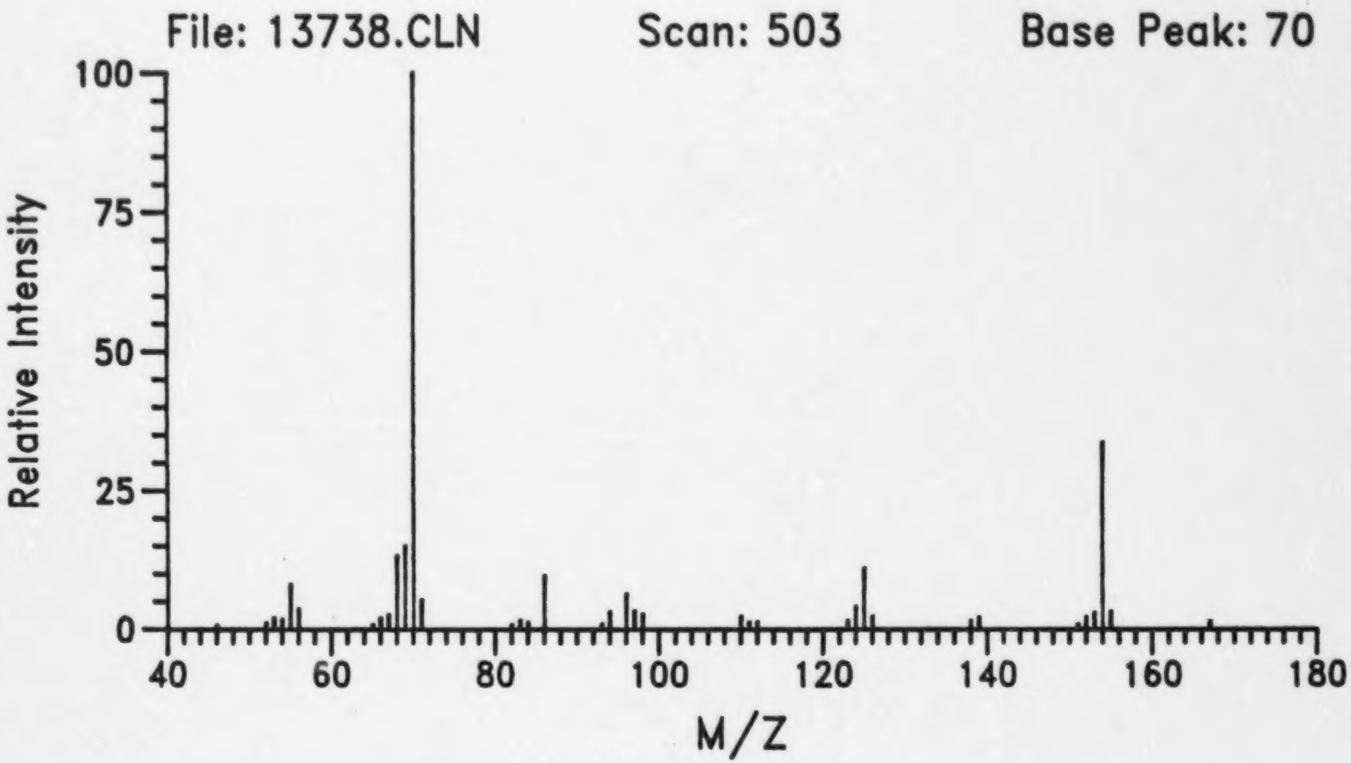


Figure A-49. Extract 13738 (base/neutral), publicly owned treatment works. Frequency of occurrence: 16.

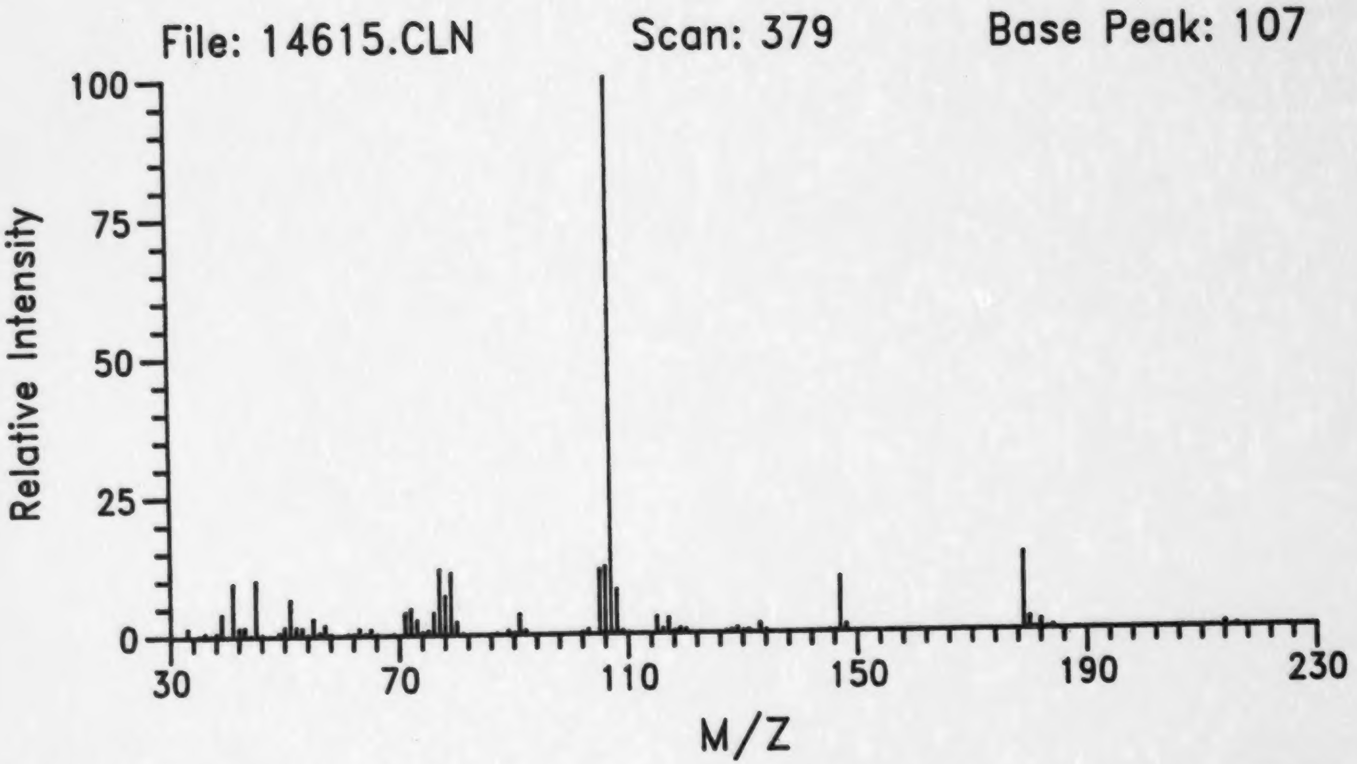


Figure A-50. Extract 14615 (Base/Neutral), Organic Chemicals Industry. Frequency of occurrence: 8.

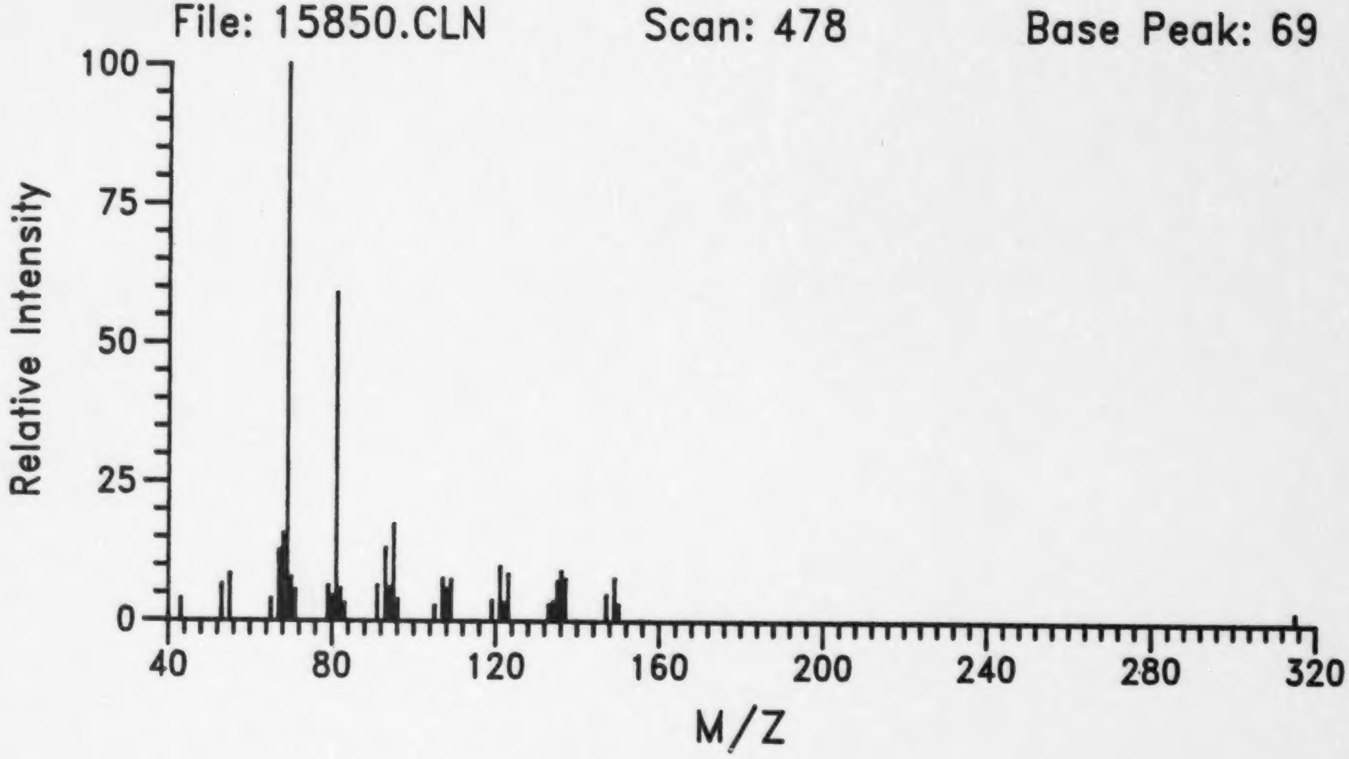


Figure A-51. Extract 15850 (Base/Neutral), Organics and Plastics Industry. Frequency of occurrence: N/A.

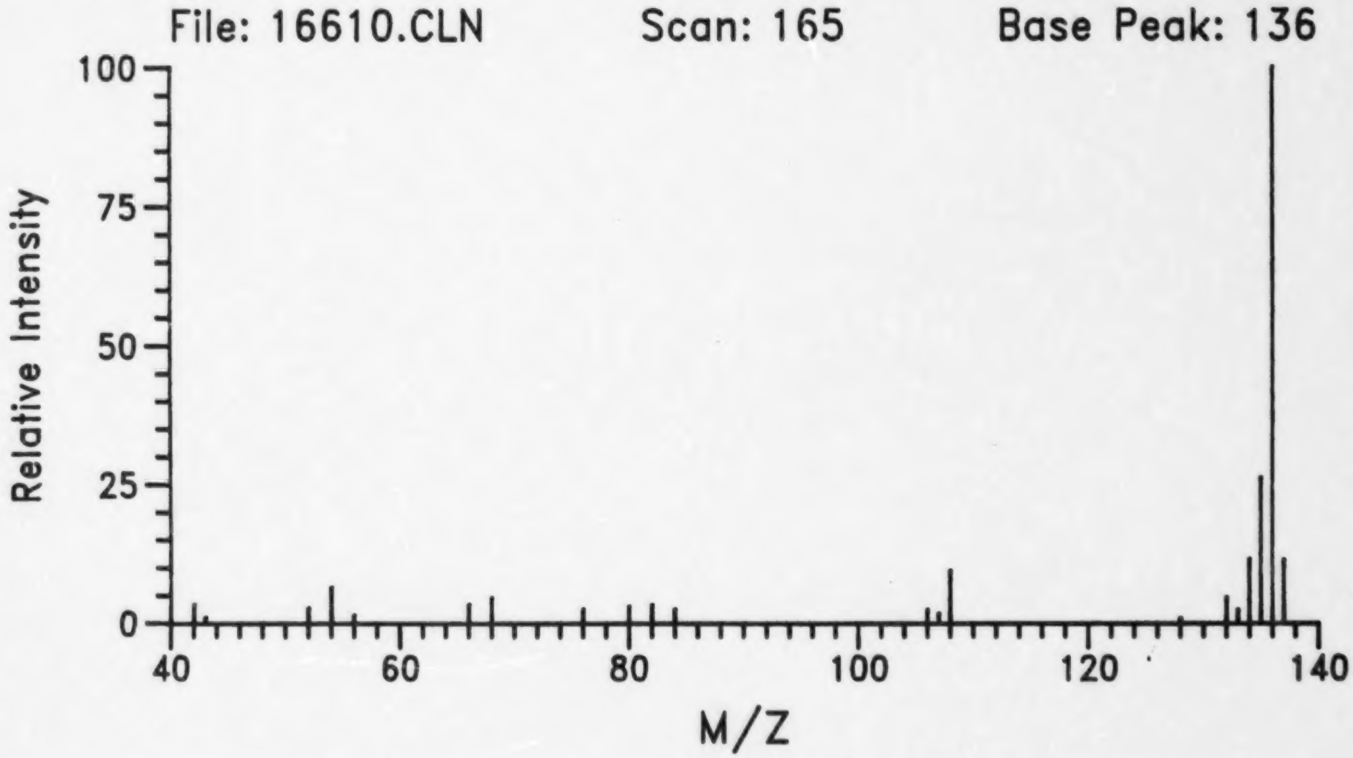


Figure A-52. Extract 16610 (Base/Neutral), Synfuels Industry. Frequency of occurrence: 30.

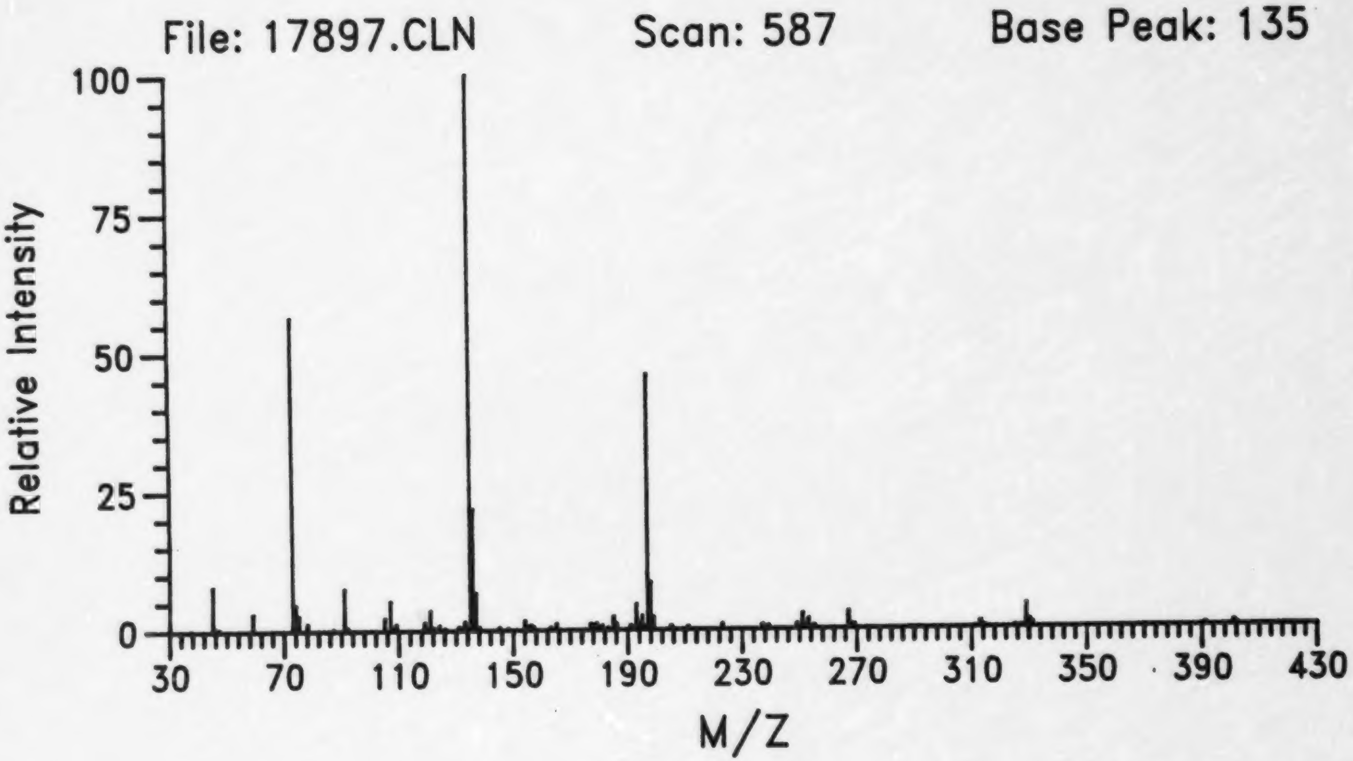


Figure A-53. Extract 17897 (Base/Neutral), Organics and Plastics Industry. Frequency of occurrence: N/A.

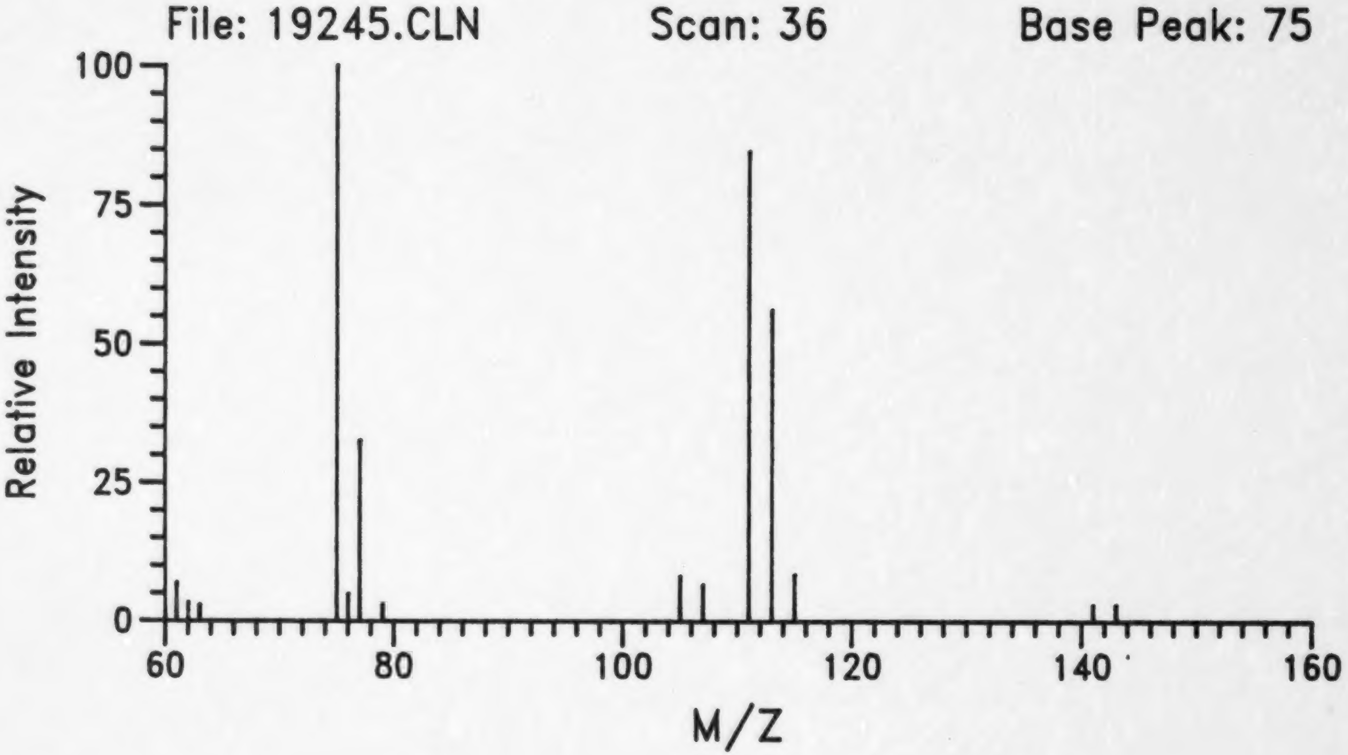


Figure A-54. Extract 19245 (Acid), Electronics Industry. Frequency of occurrence: 8.

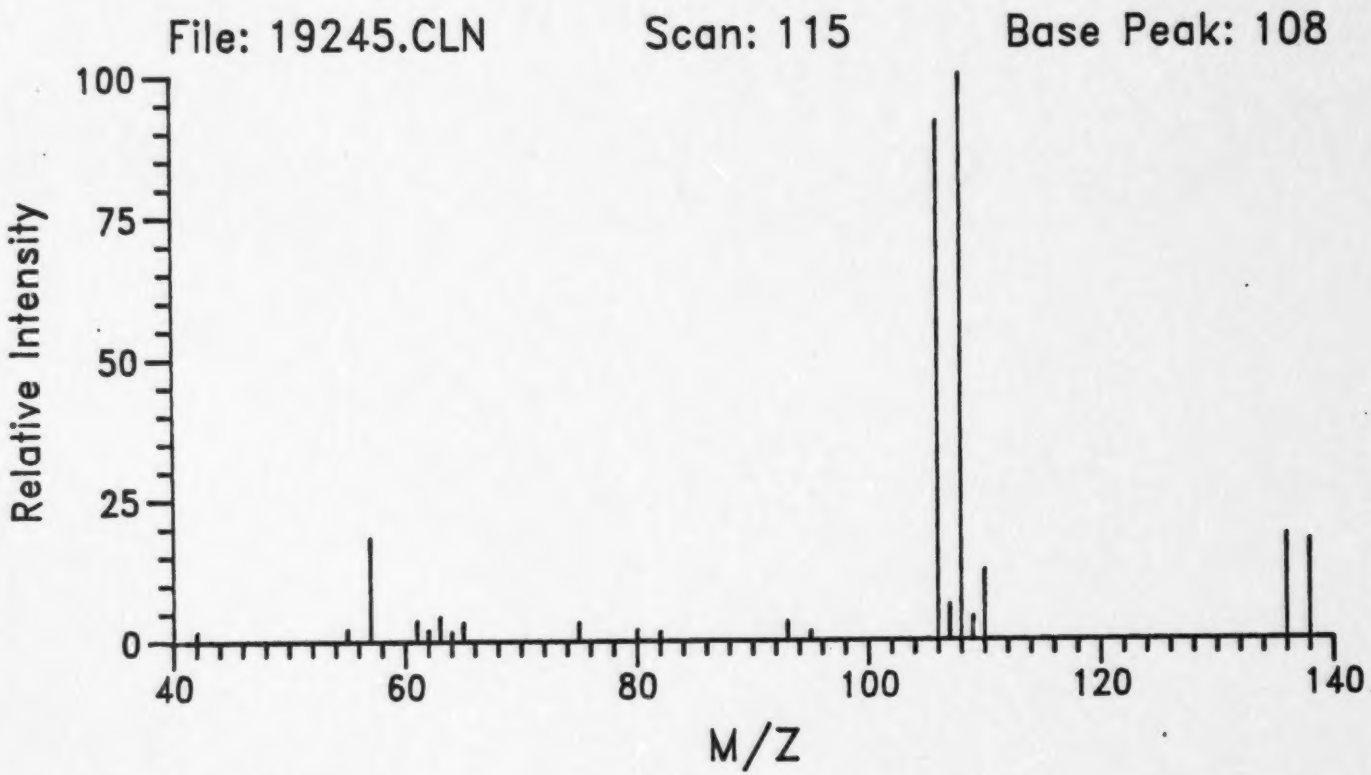


Figure A-55. Extract 19245 (Acid), Electronics Industry. Frequency of occurrence: 9.

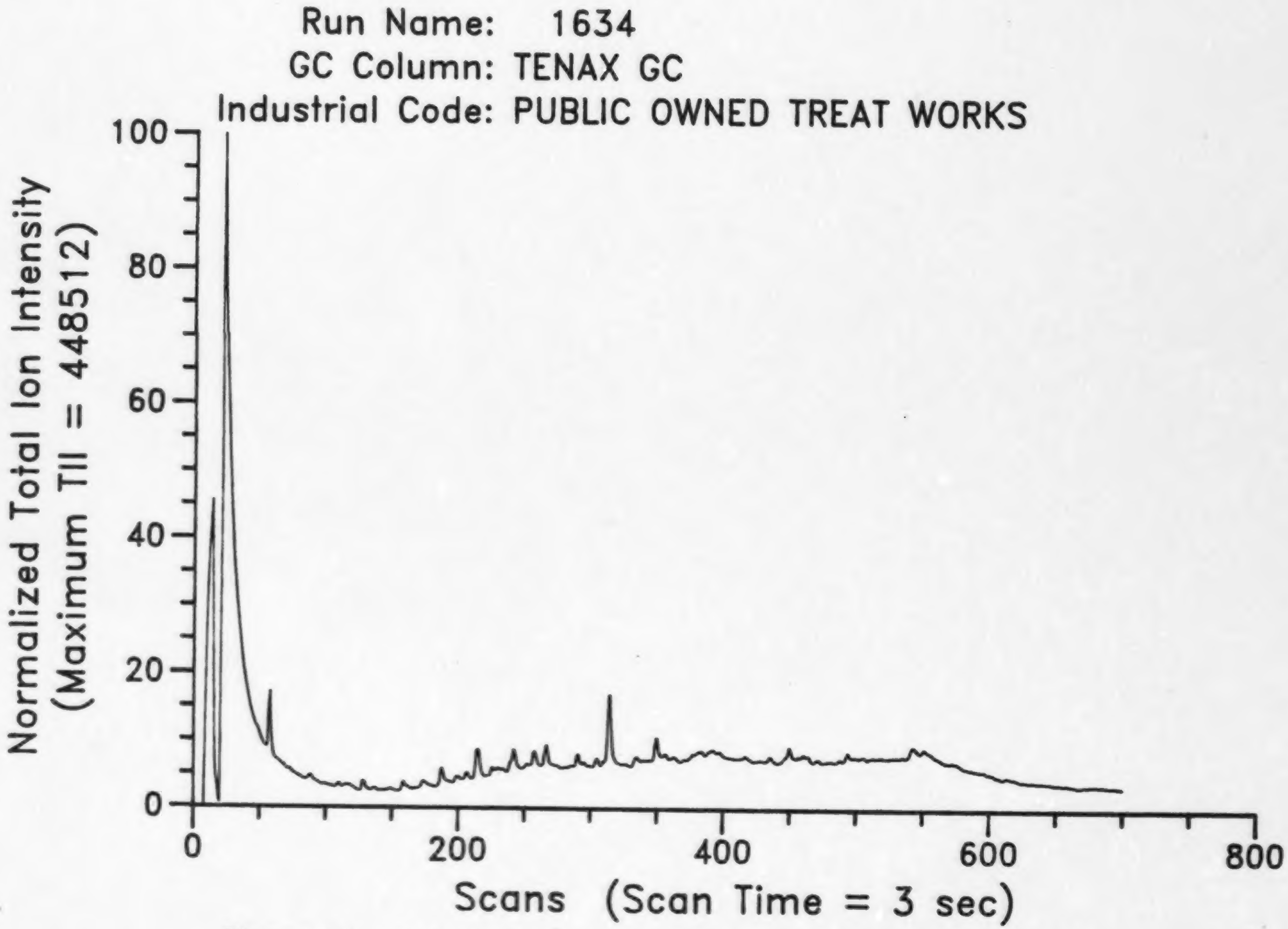


Figure B-1. Packed column chromatogram, extract 1634 (acid), publicly owned treatment works.

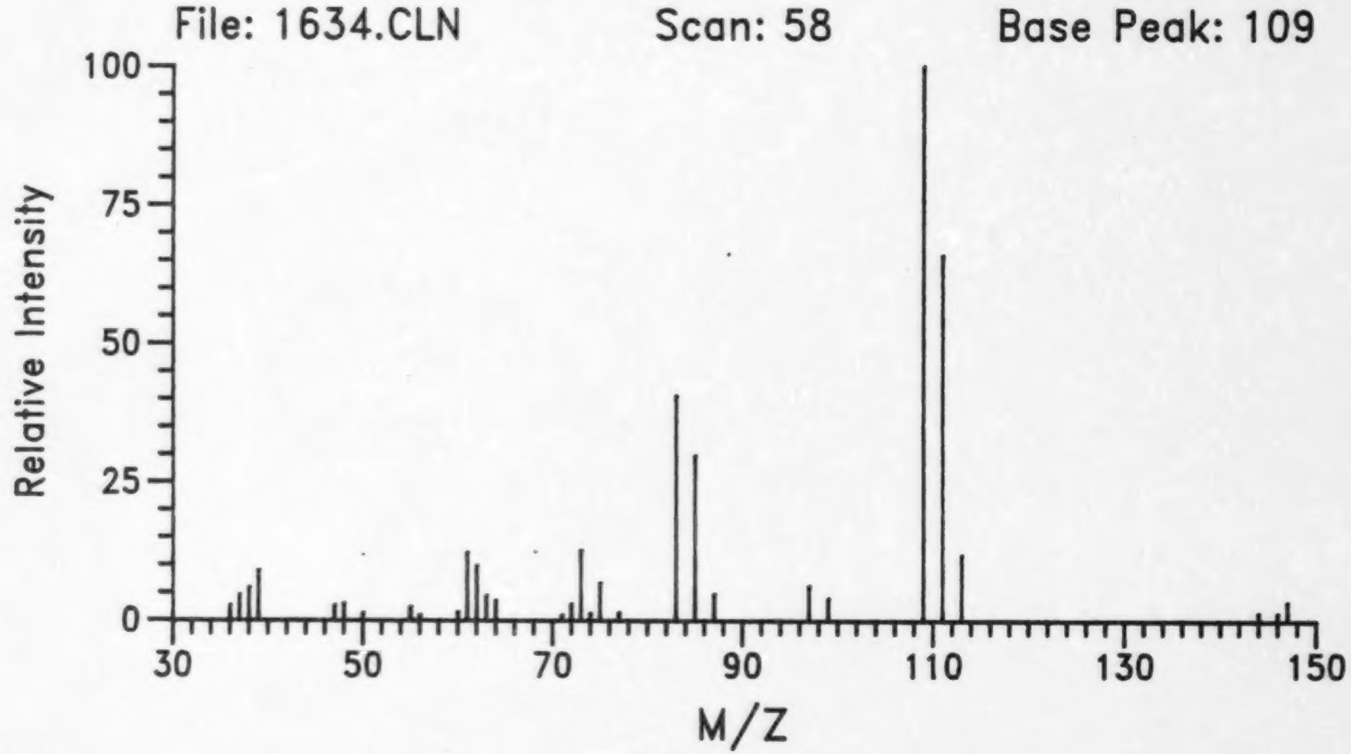


Figure B-2. Extract 1634 (acid), publicy owned treatment works.
Frequency of occurrence: 13.

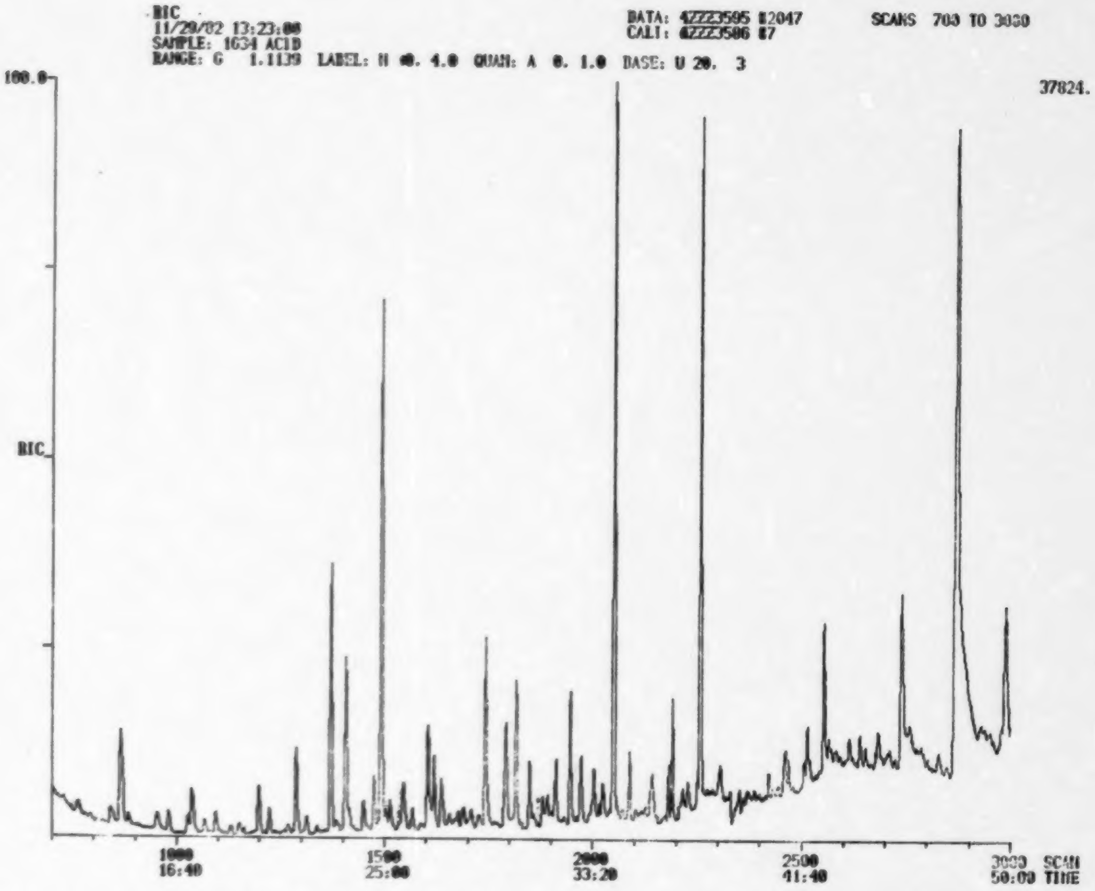


Figure B-3. Capillary chromatogram of extract 1634 (acid), publicly owned treatment works.

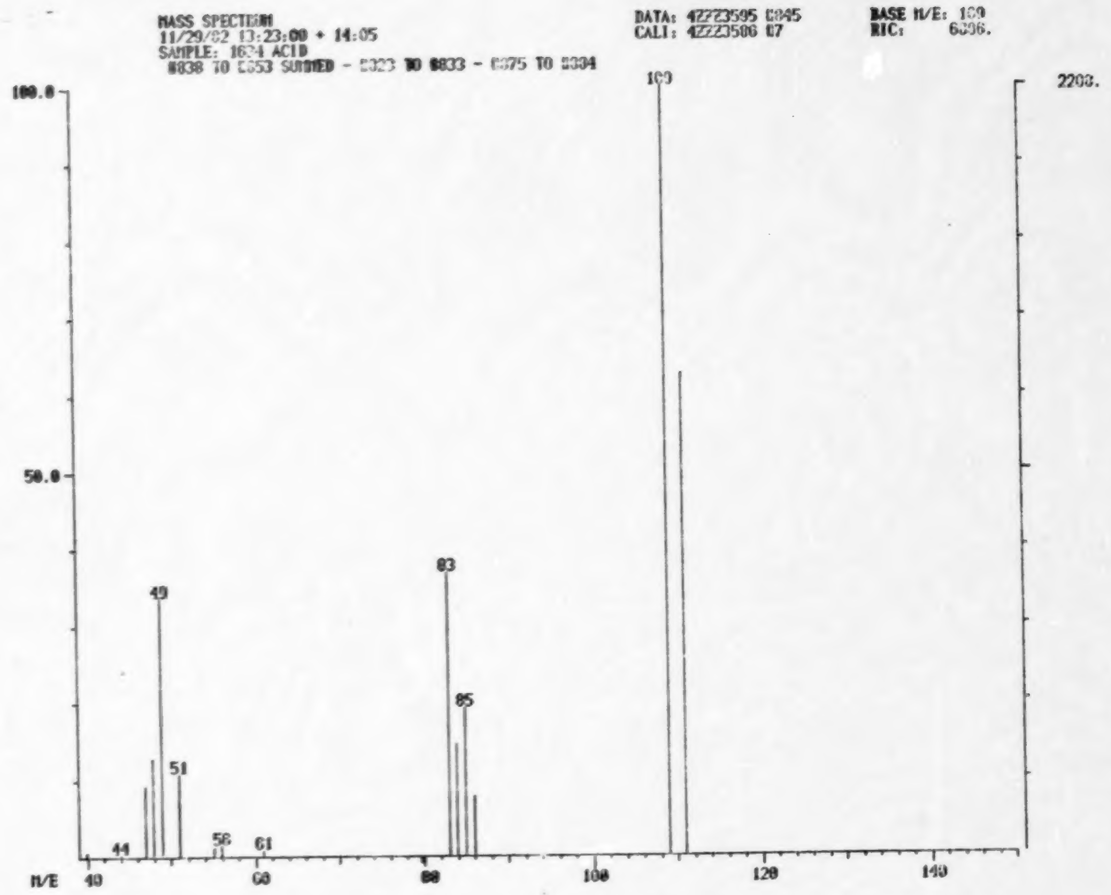


Figure B-4. Mass spectrum of EPA compound of interest, extract 1634 (acid), publicly owned treatment works, scan #845.

Figure B-5. Conclusions, Extract 1634 (acid)

The compound of interest is at least a trichloropropene; it may contain more than three chlorine atoms. Positive and negative chemical ionization (methane) shows no reaction. Very few reference spectra or standards are available. The ions at mass 109/111 are representative of a two-chlorine-containing fragment, but no molecular ion information can be obtained.

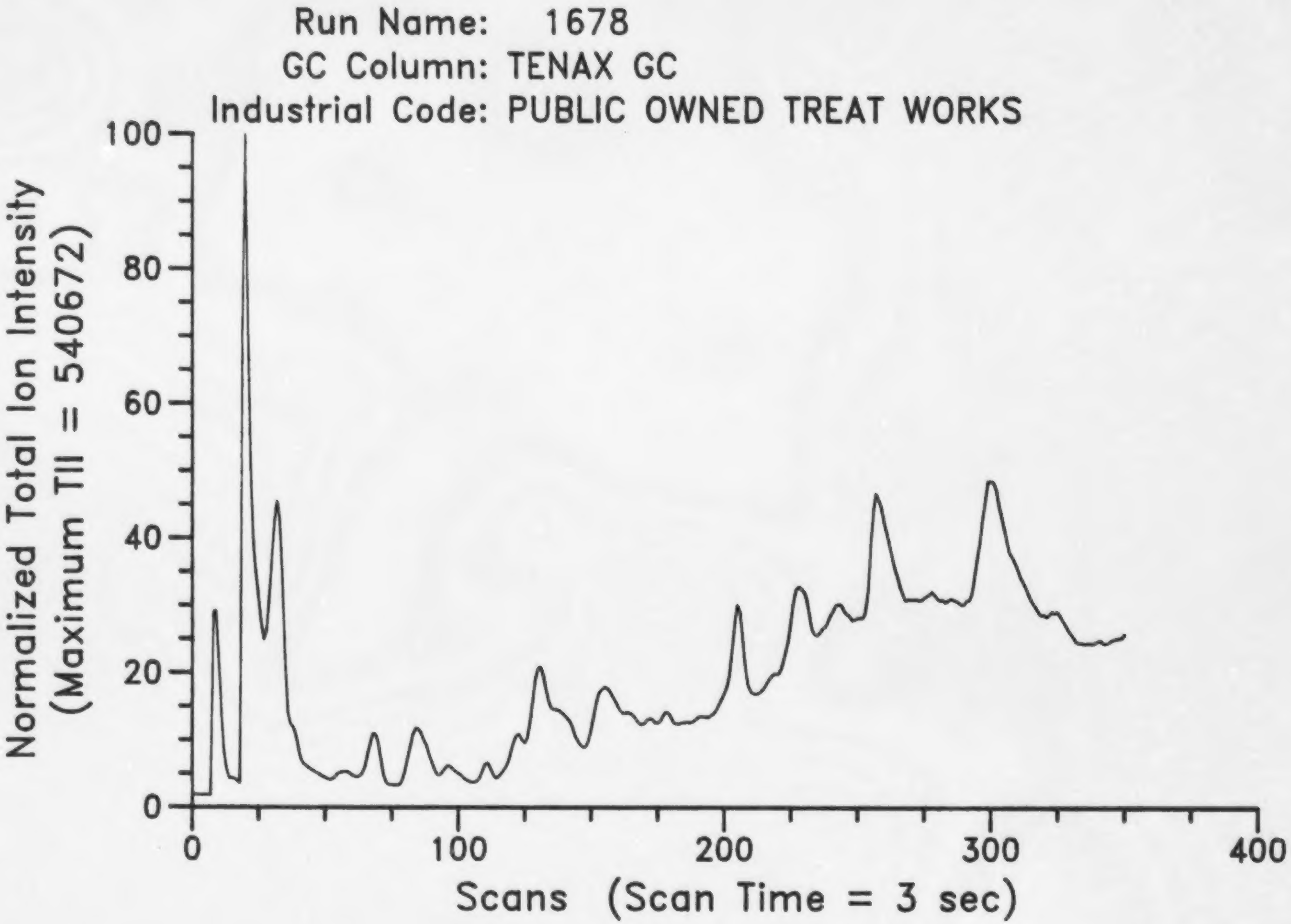


Figure B-6. Packed column chromatogram of extract 1678 (acid), publicly owned treatment works.

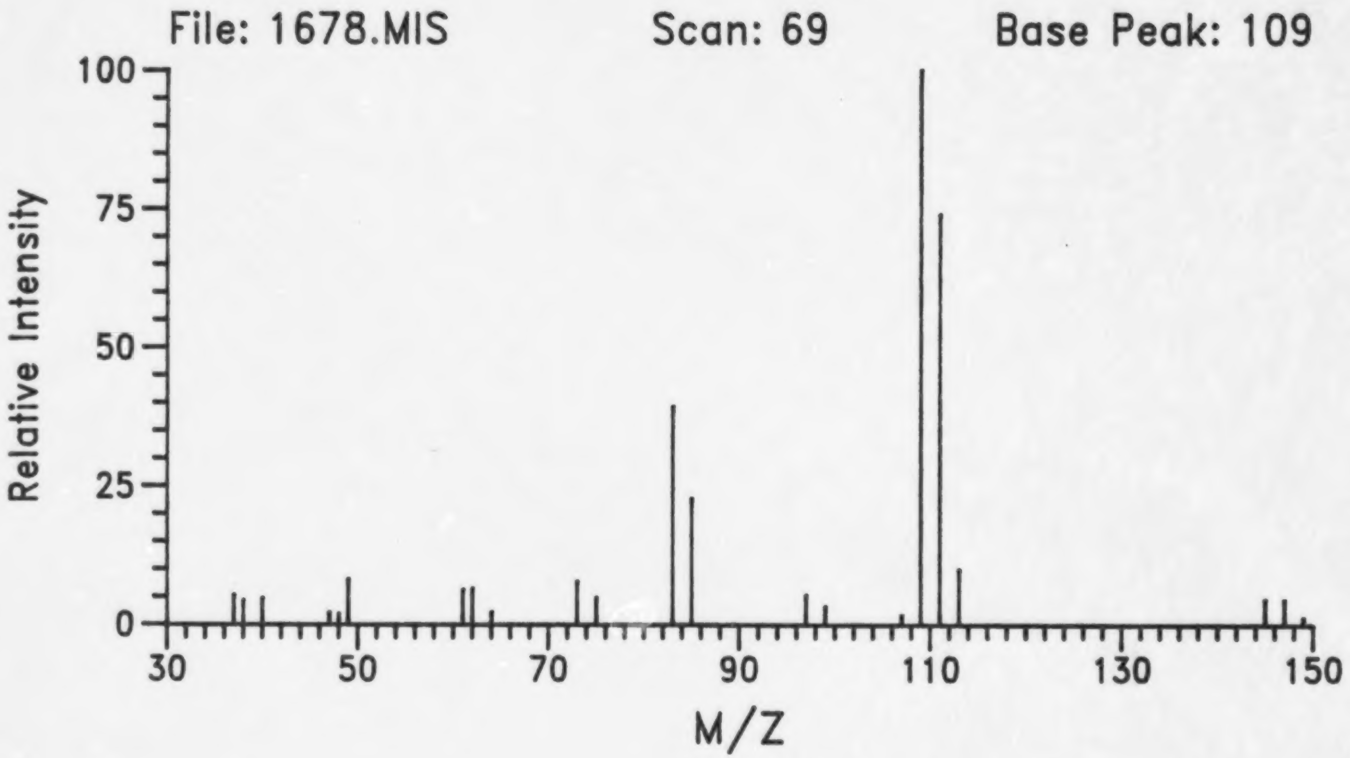


Figure B-7. Extract 1678 (acid), publicly owned treatment works. Frequency of occurrence: 7.

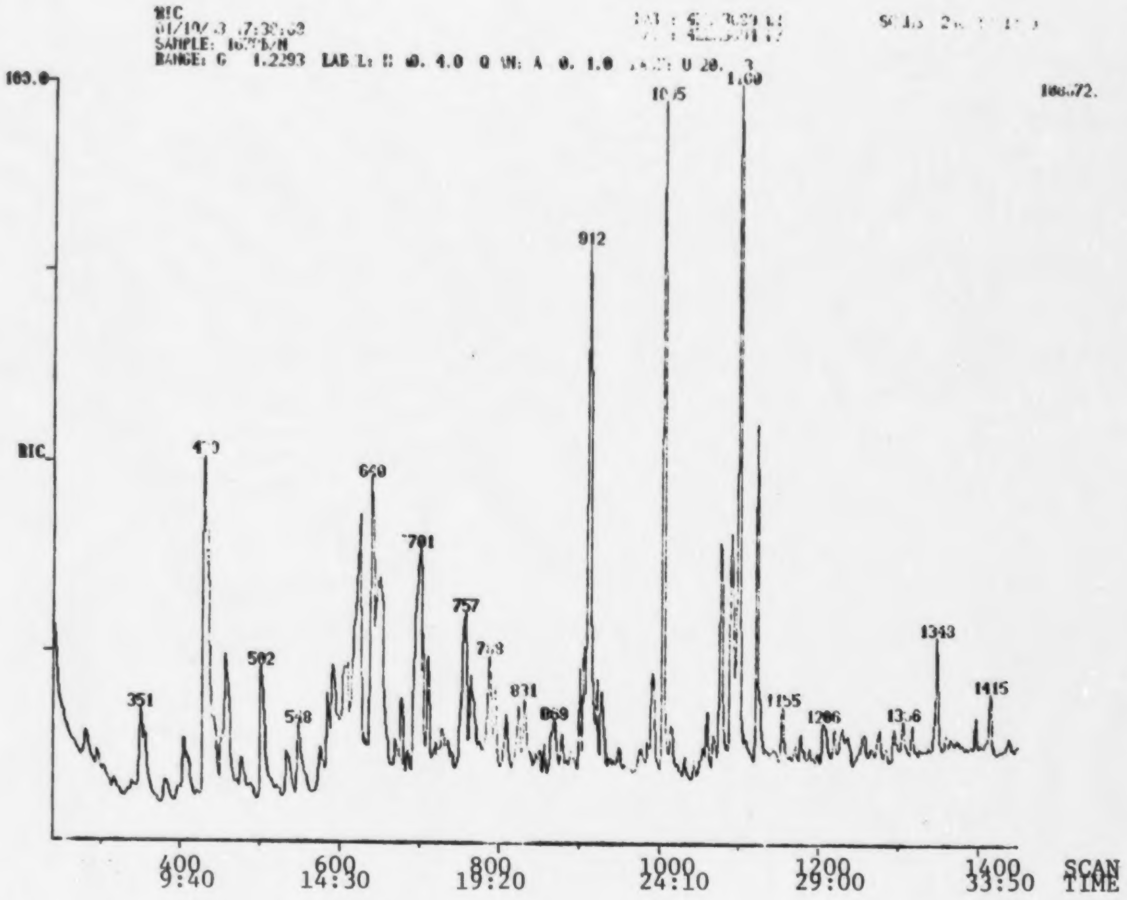


Figure B-8. Capillary chromatogram, extract 1678 (acid), publicly owned treatment works.

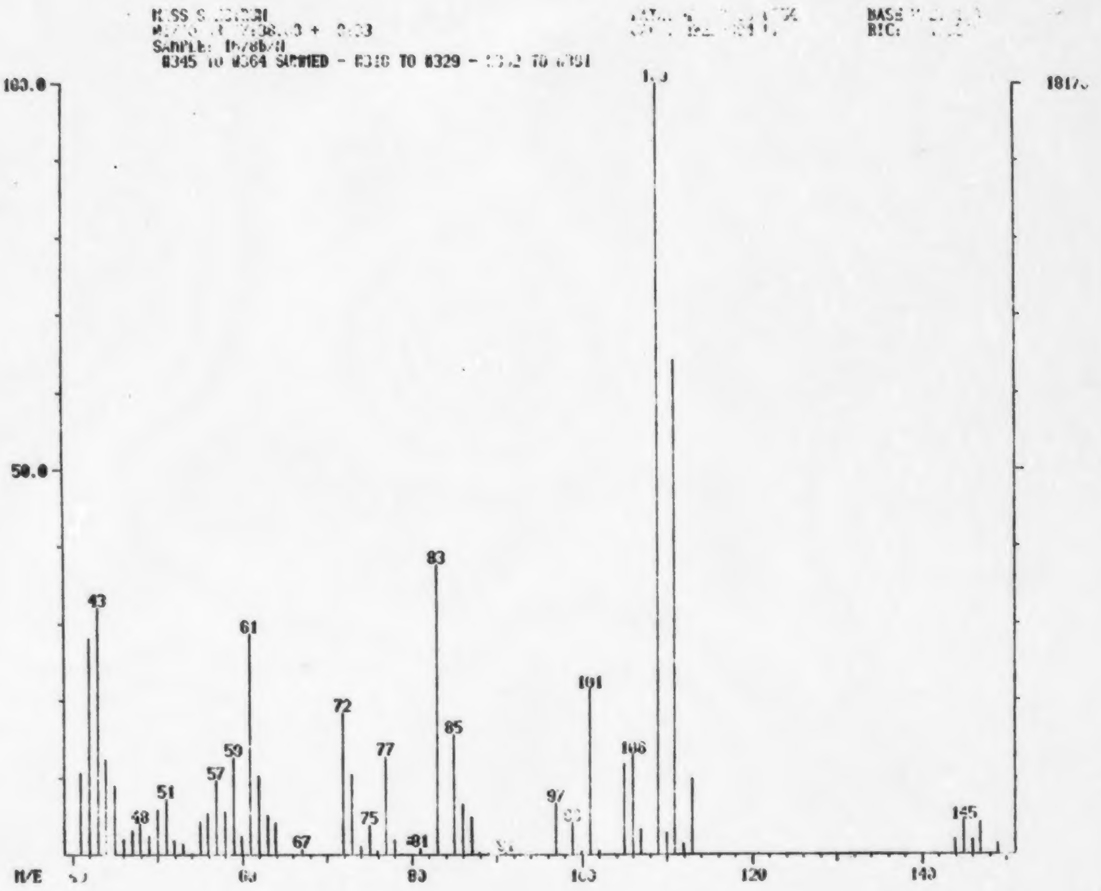


Figure B-9. Mass spectrum of EPA compound of interest, extract 1678 (acid), publicly owned treatment works, scan #355.

Figure B-10. Conclusions, Extract 1678 (acid)

The compound of interest is at least a trichloropropene; it may contain more than three chlorine atoms. Positive and negative chemical ionization (methane) shows no reaction. Very few reference spectra or standards are available. The ions at mass 109/111 are representative of a two-chlorine-containing fragment, but no molecular ion information can be obtained.

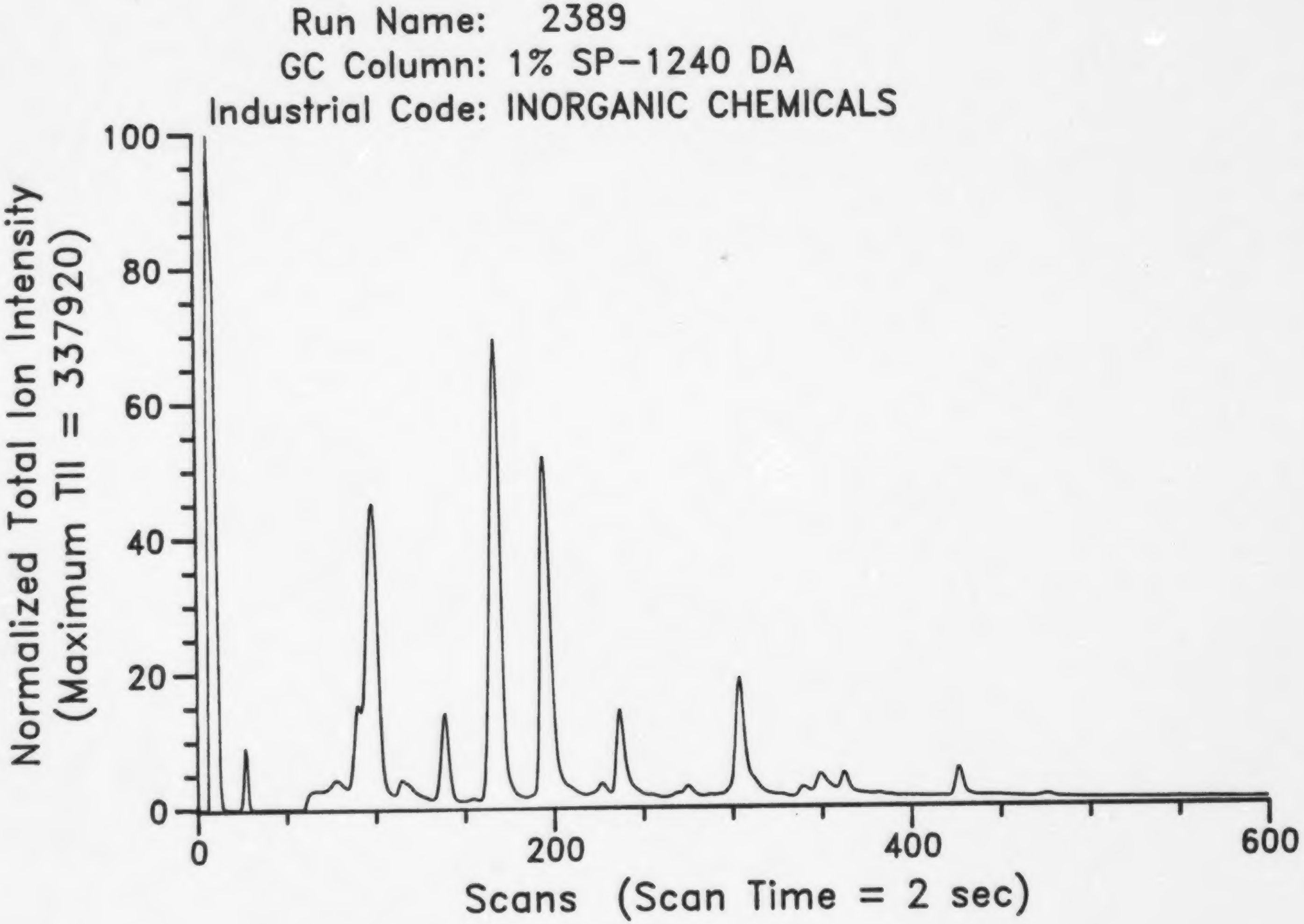


Figure B-11. Packed column chromatogram, extract 2389 (acid), inorganic chemicals industry.

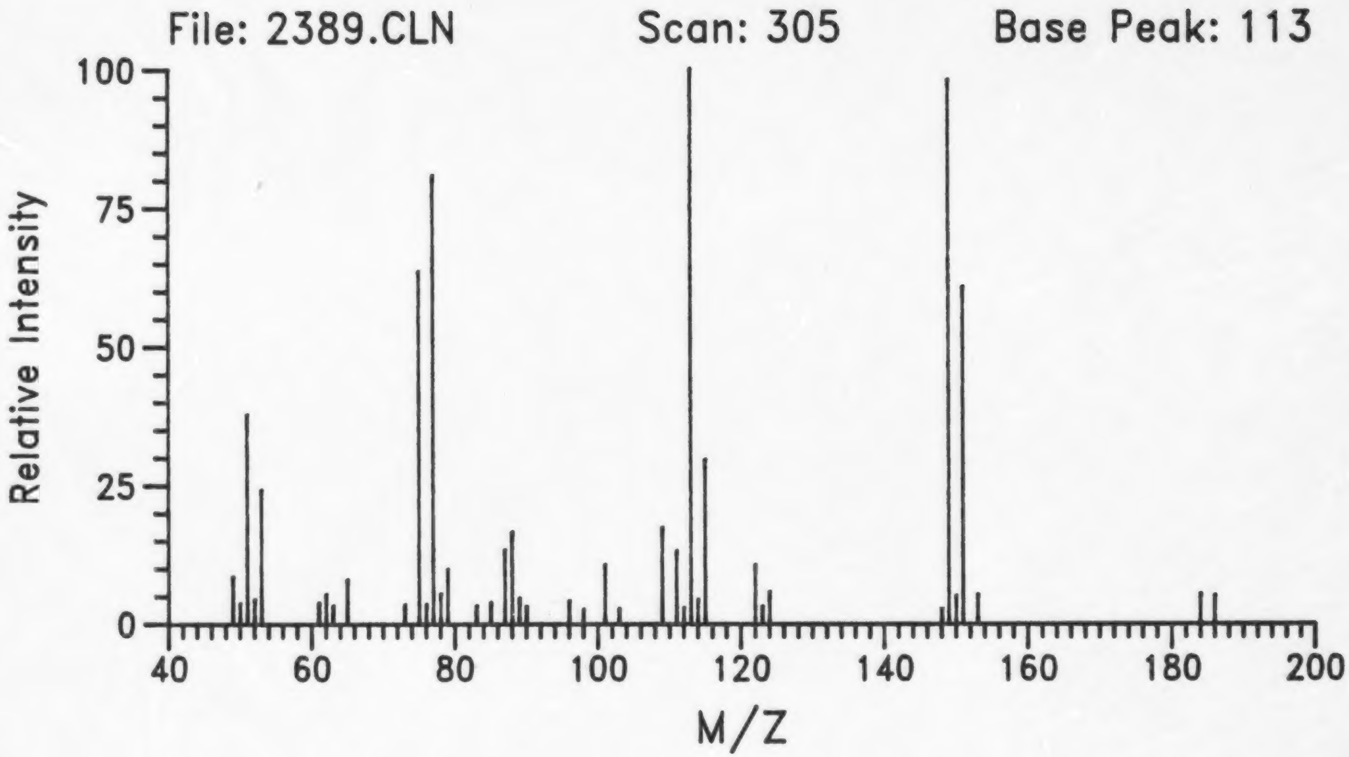


Figure B-12. Extract 2389 (acid), inorganic chemicals industry.
Frequency of occurrence: 6.

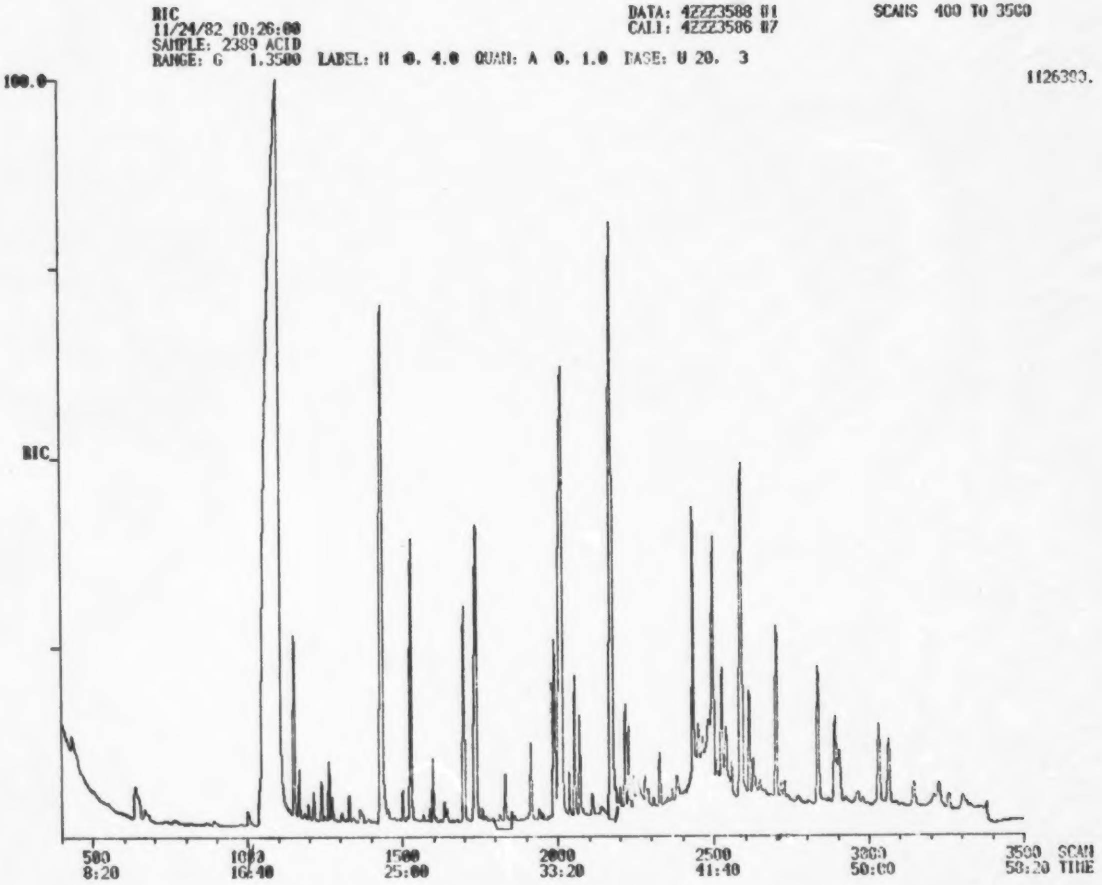


Figure B-13. Capillary chromatogram, extract 2389 (acid), inorganic chemicals industry.

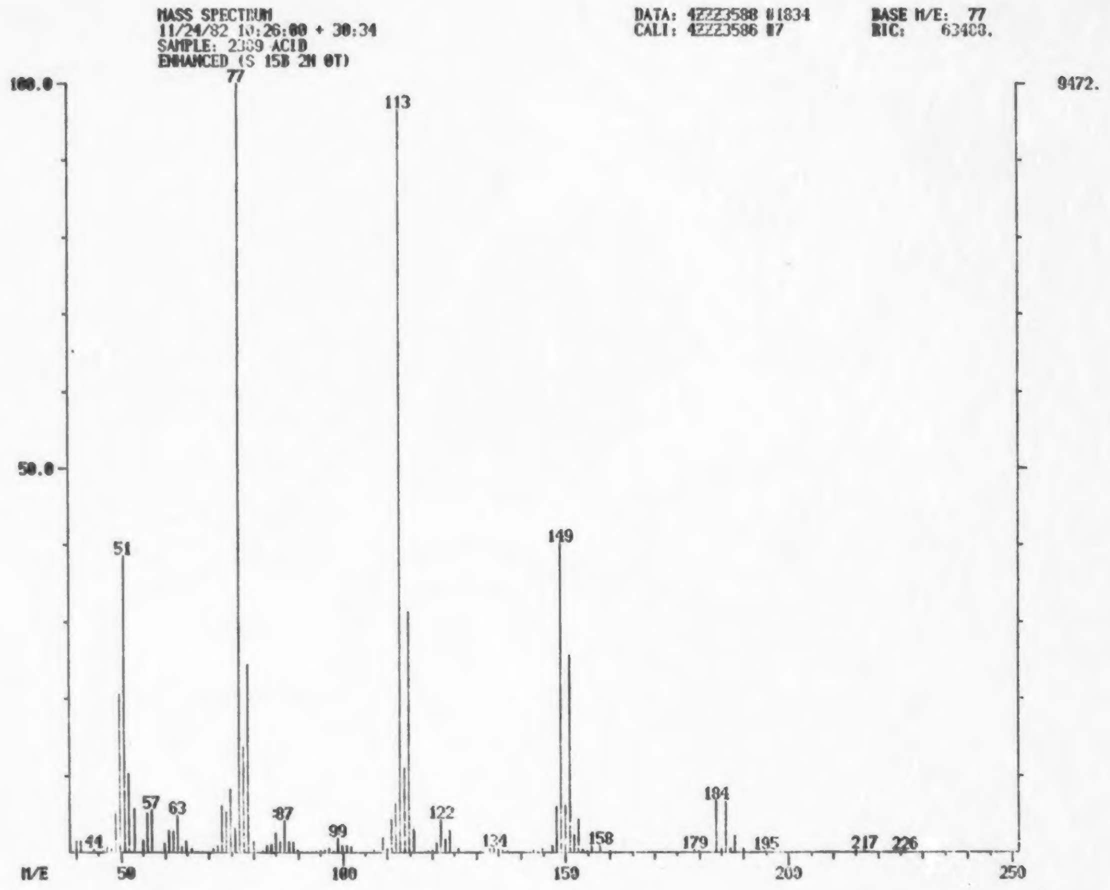


Figure B-14. Mass spectrum of EPA compound of interest, extract 2389 (acid) inorganic chemicals industry, scan #1834.

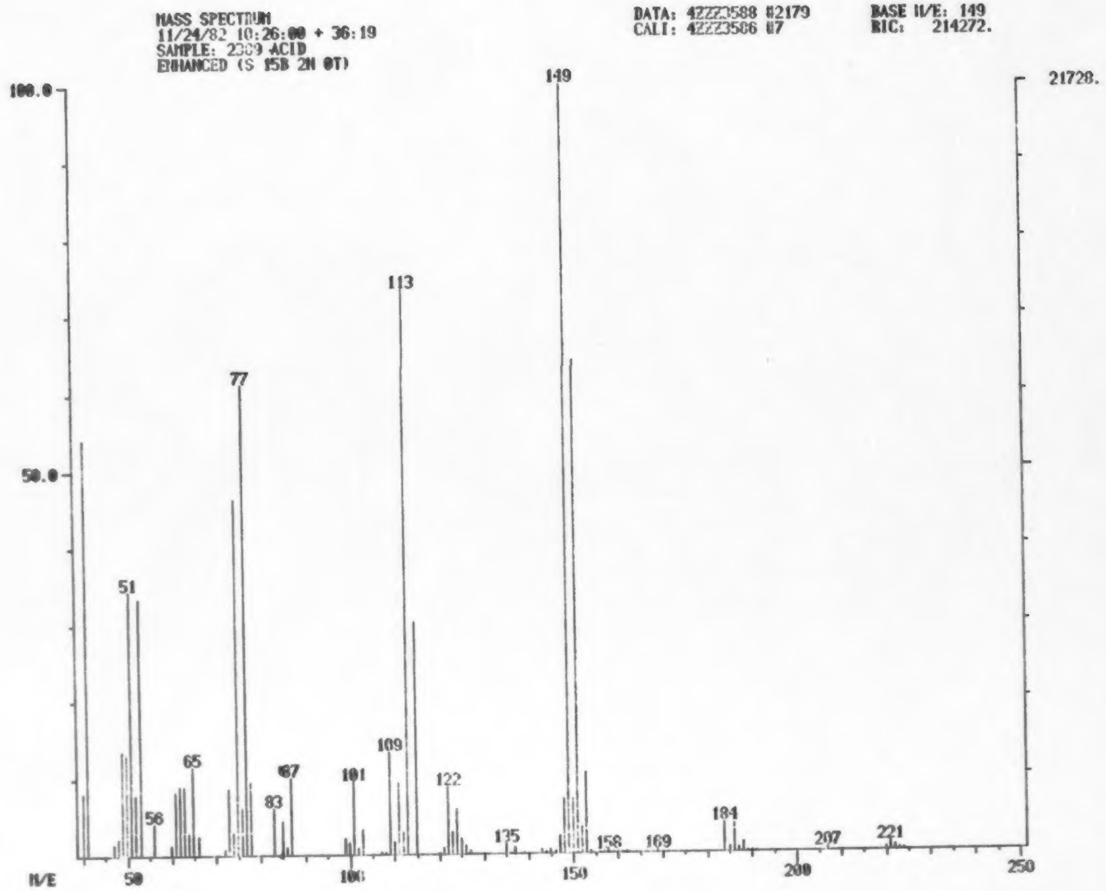


Figure B-15. Mass spectrum of EPA compound of interest, extract 2389 (acid) inorganic chemicals industry, scan #2179.

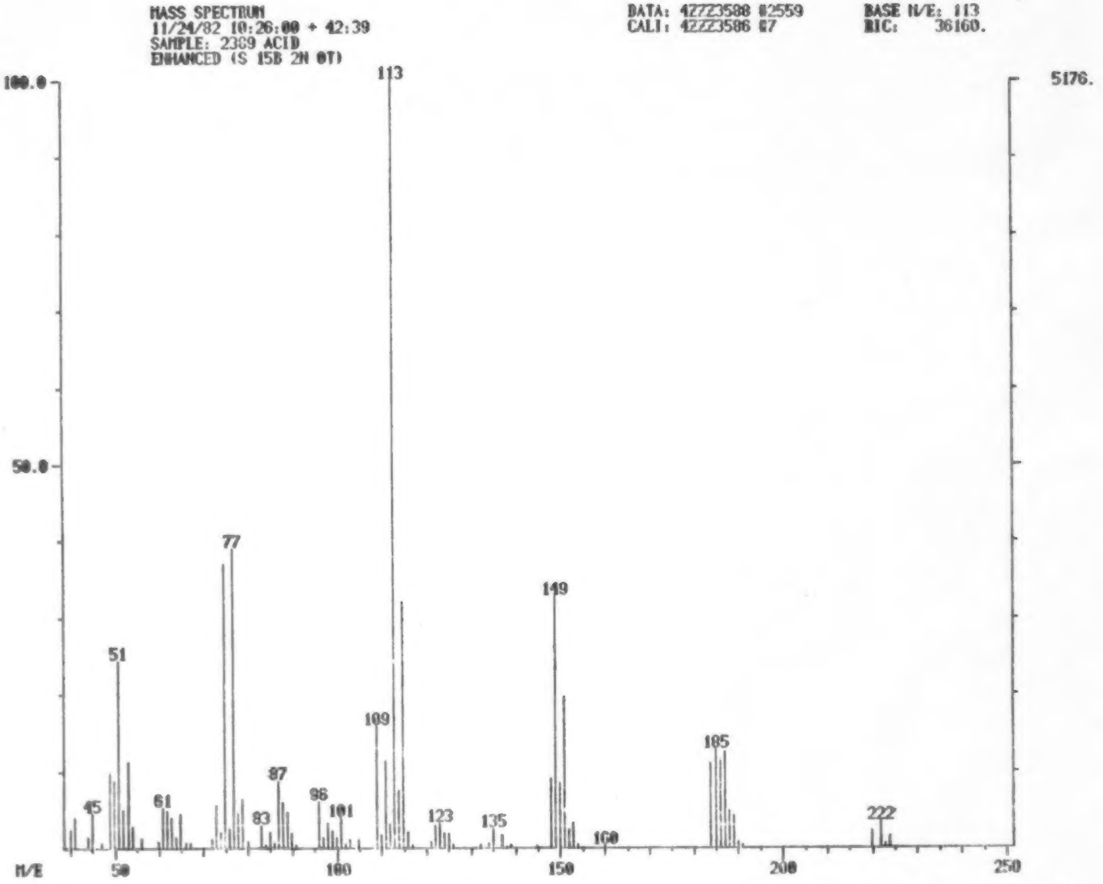


Figure B-16. Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2559.

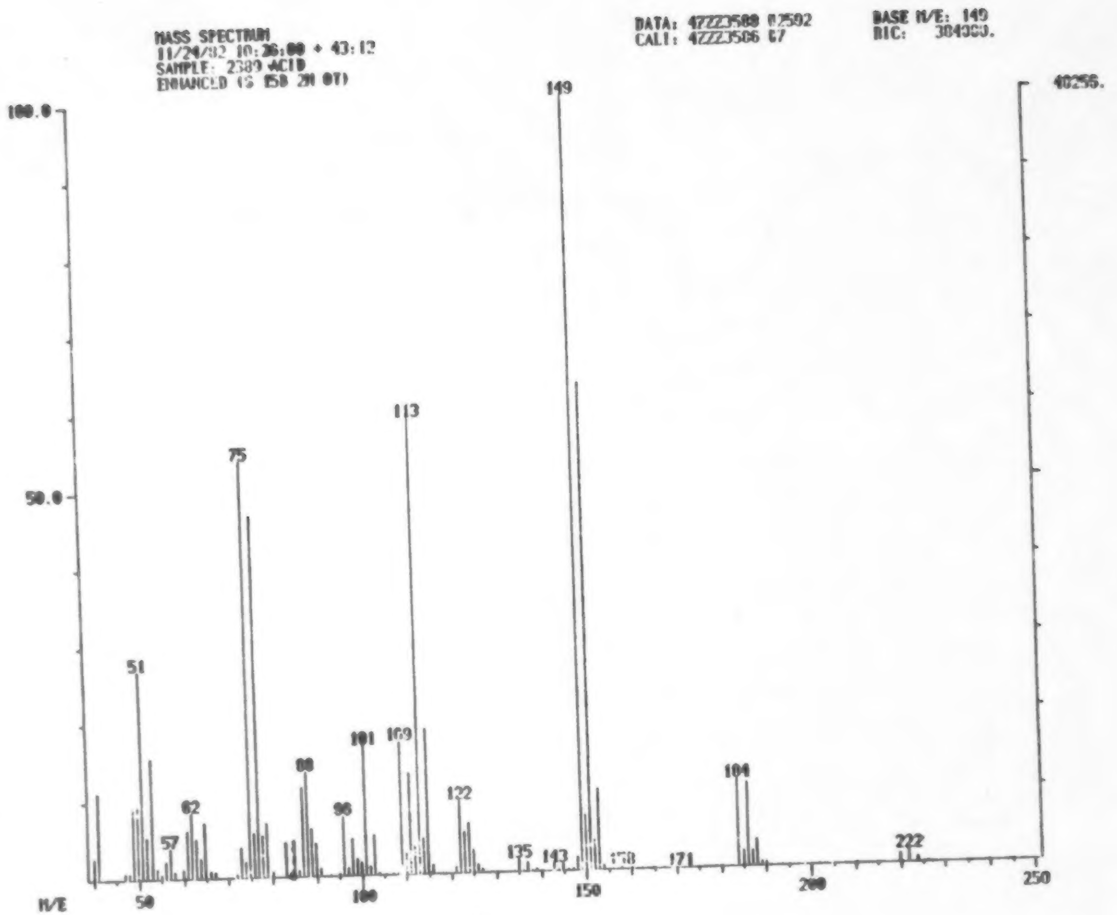


Figure B-17. Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2592.

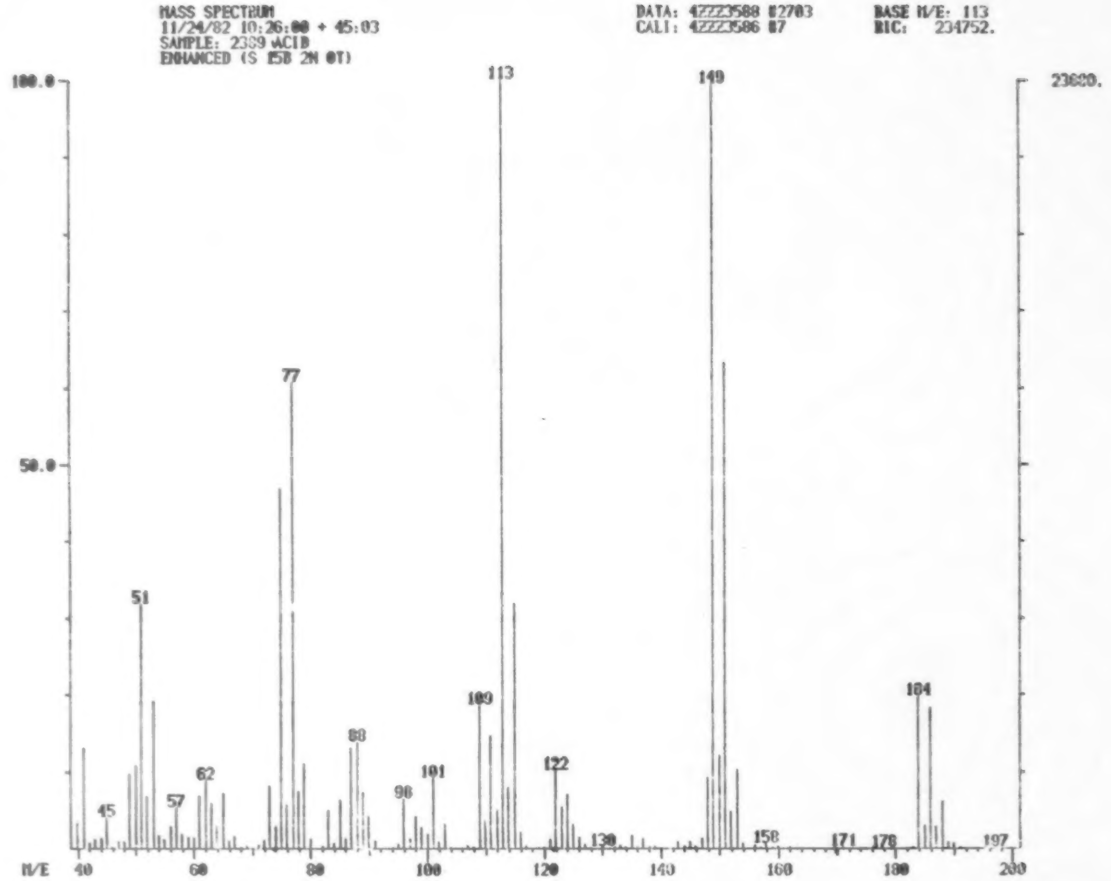


Figure B-18. Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2703.

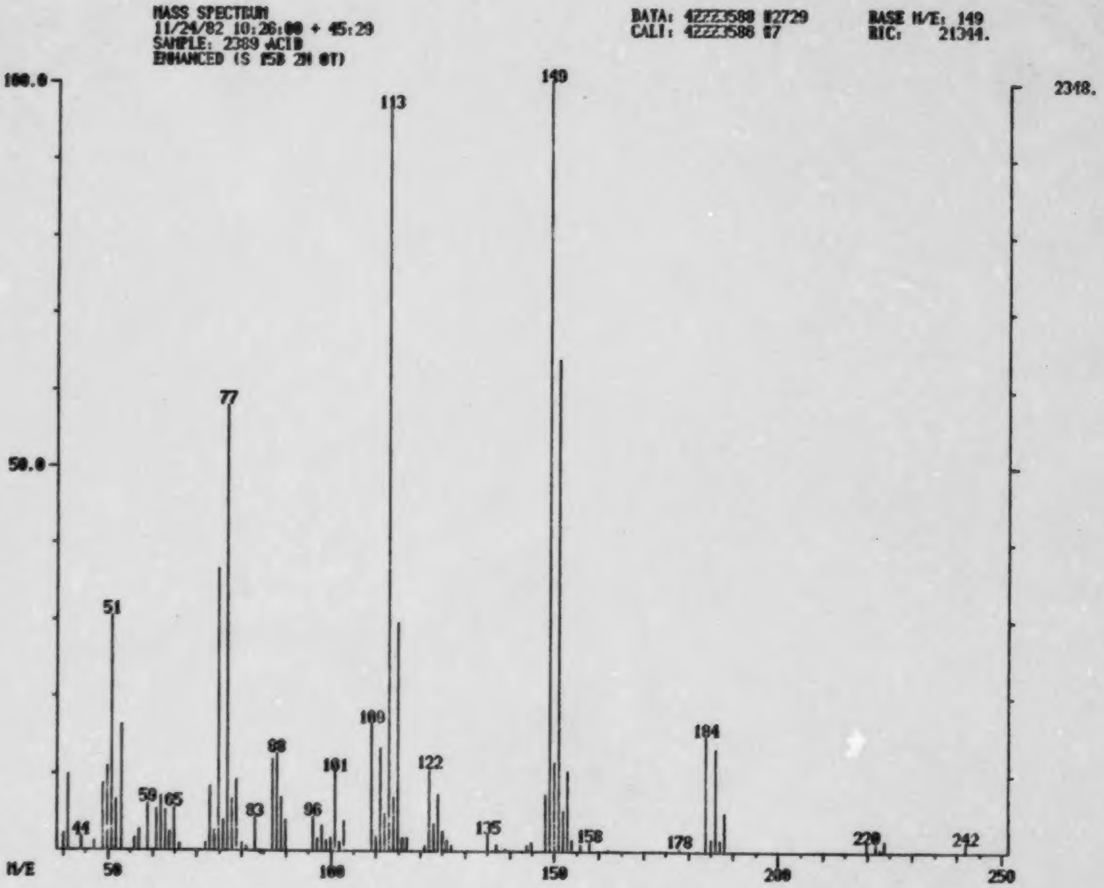


Figure B-19. Mass spectrum of EPA compound of interest, extract 2389 (acid), inorganic chemicals industry, scan #2729.

Figure B-20. Conclusion, Extract 2389 (acid)

The compounds of interest appear to be a family of trichlorocyclohexenes, as substantiated by accurate mass measurement. Identification as a class is relatively straightforward. Isomer speciation would require either isolation for NMR analysis or use of standards (not commercially available) to establish relative chromatographic retention times. There are differences in relative abundances of the different ions. Some spectra show the presence of a four-chlorine isotope cluster at $\underline{m}/\underline{z}$ 220-226, indicating four chlorine atoms on a saturated cyclohexane ring.

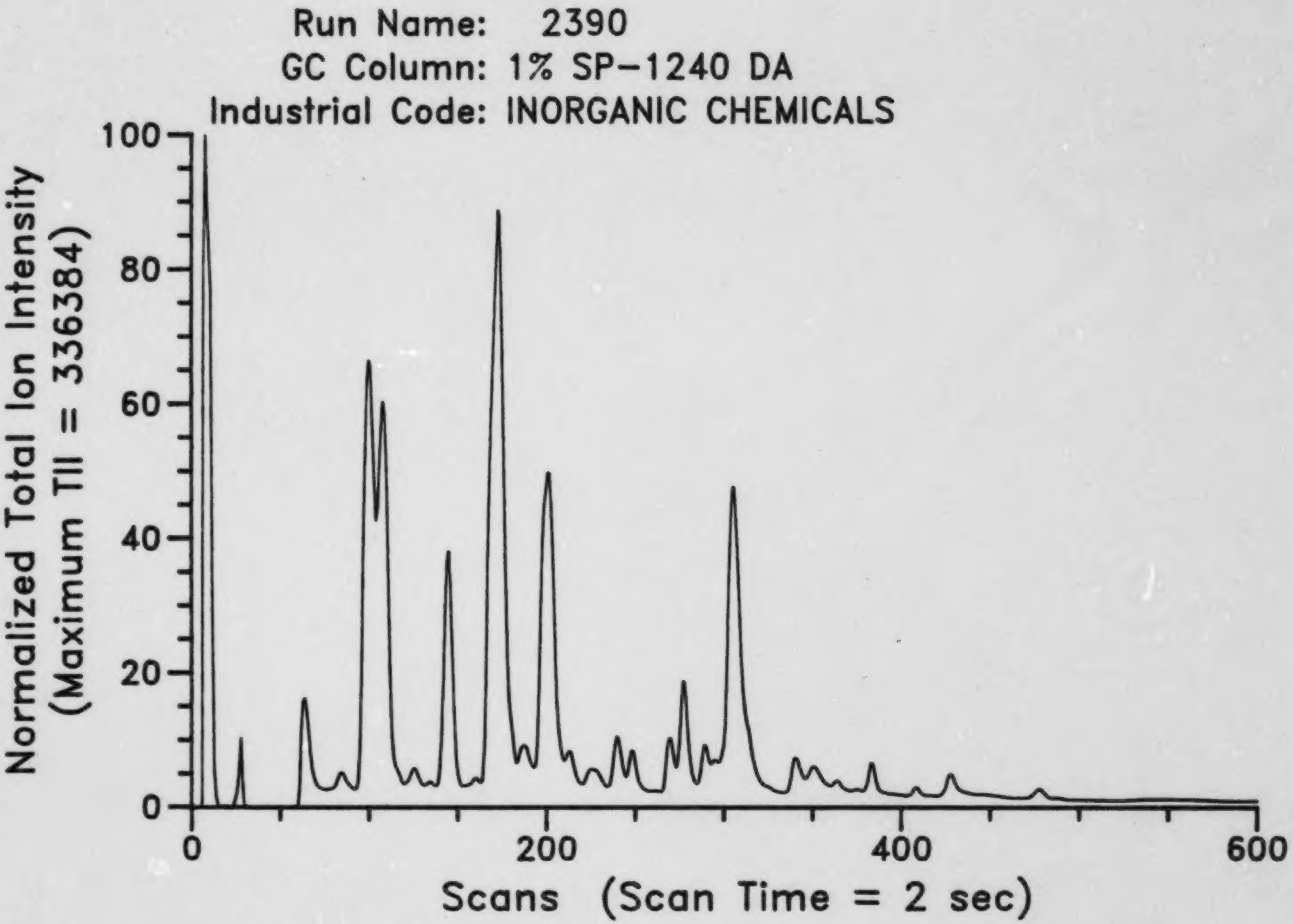


Figure B-21. Packed column chromatogram, extract 2390 (acid), inorganic chemicals industry.

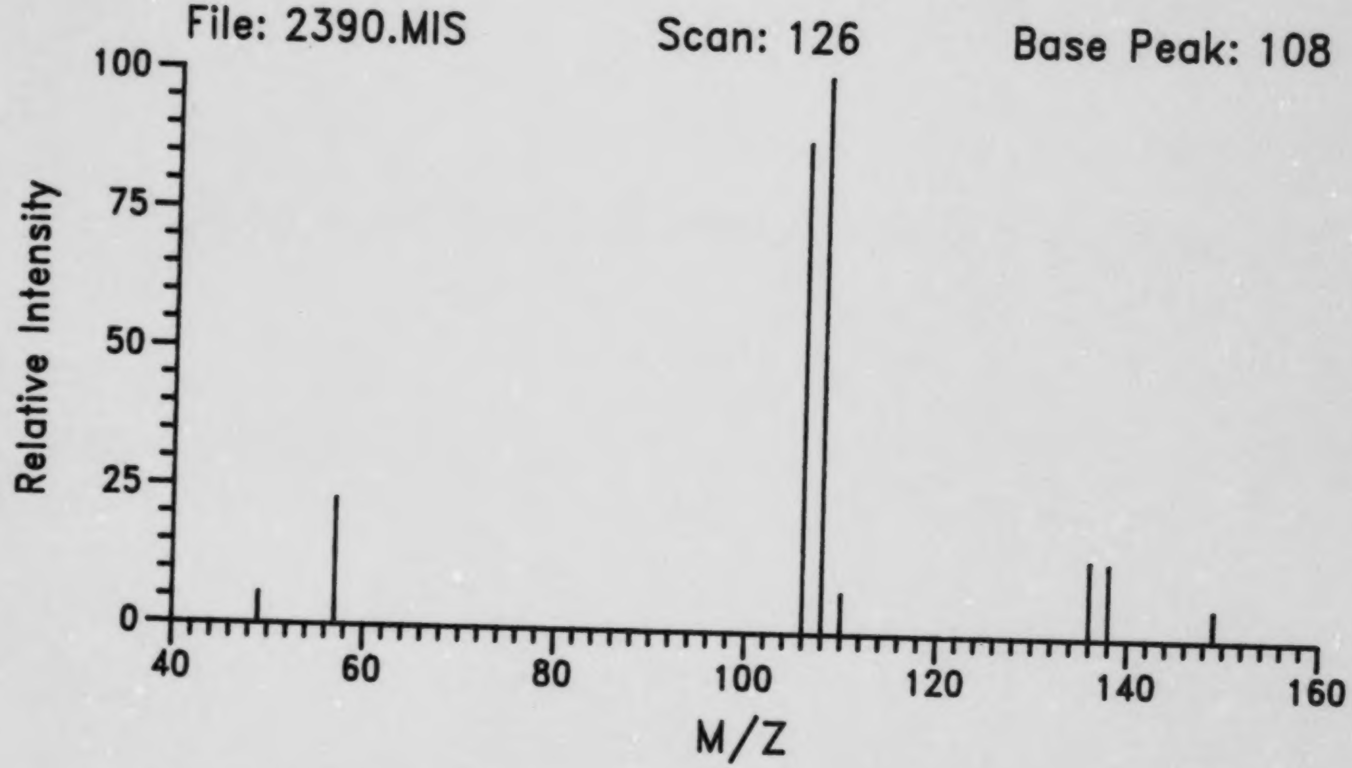


Figure B-22. Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 7.

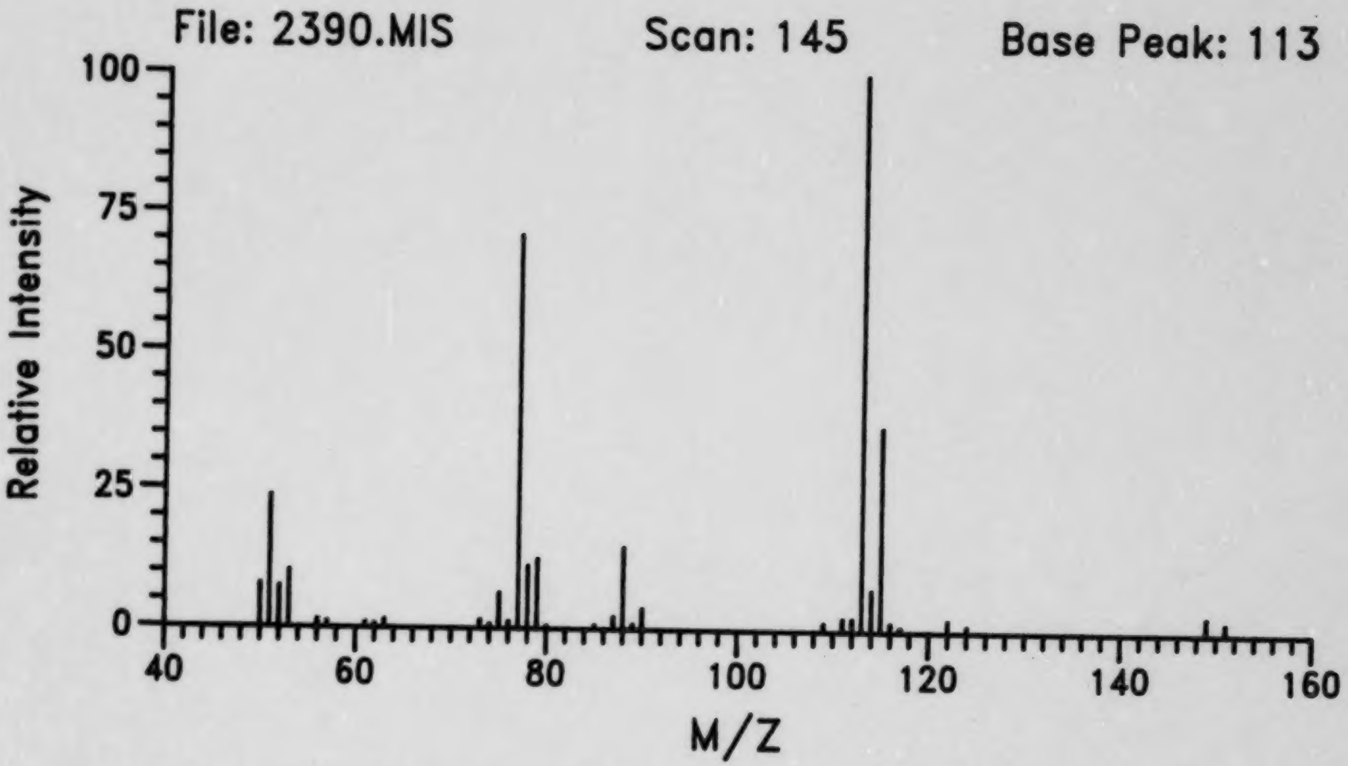


Figure B-23. Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 6.

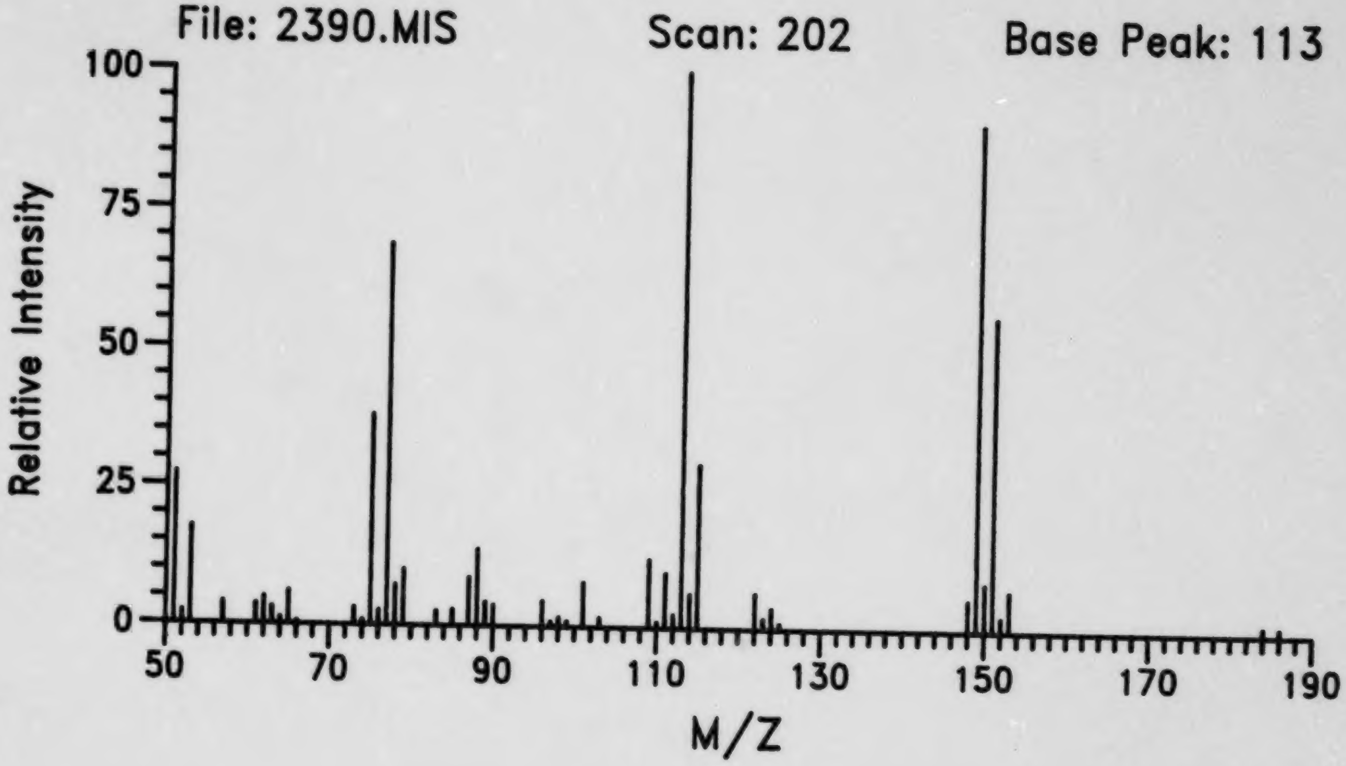


Figure B-24. Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 9.

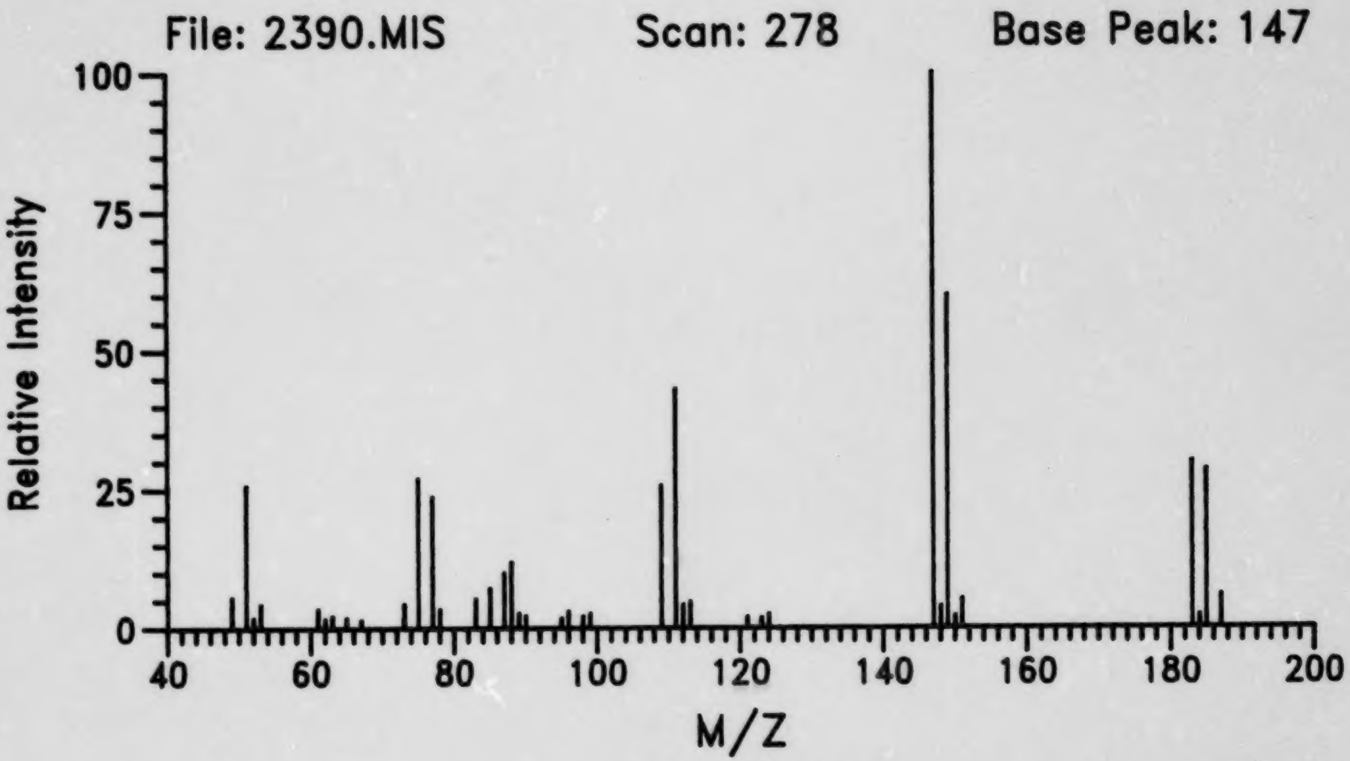


Figure B-25. Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 9.

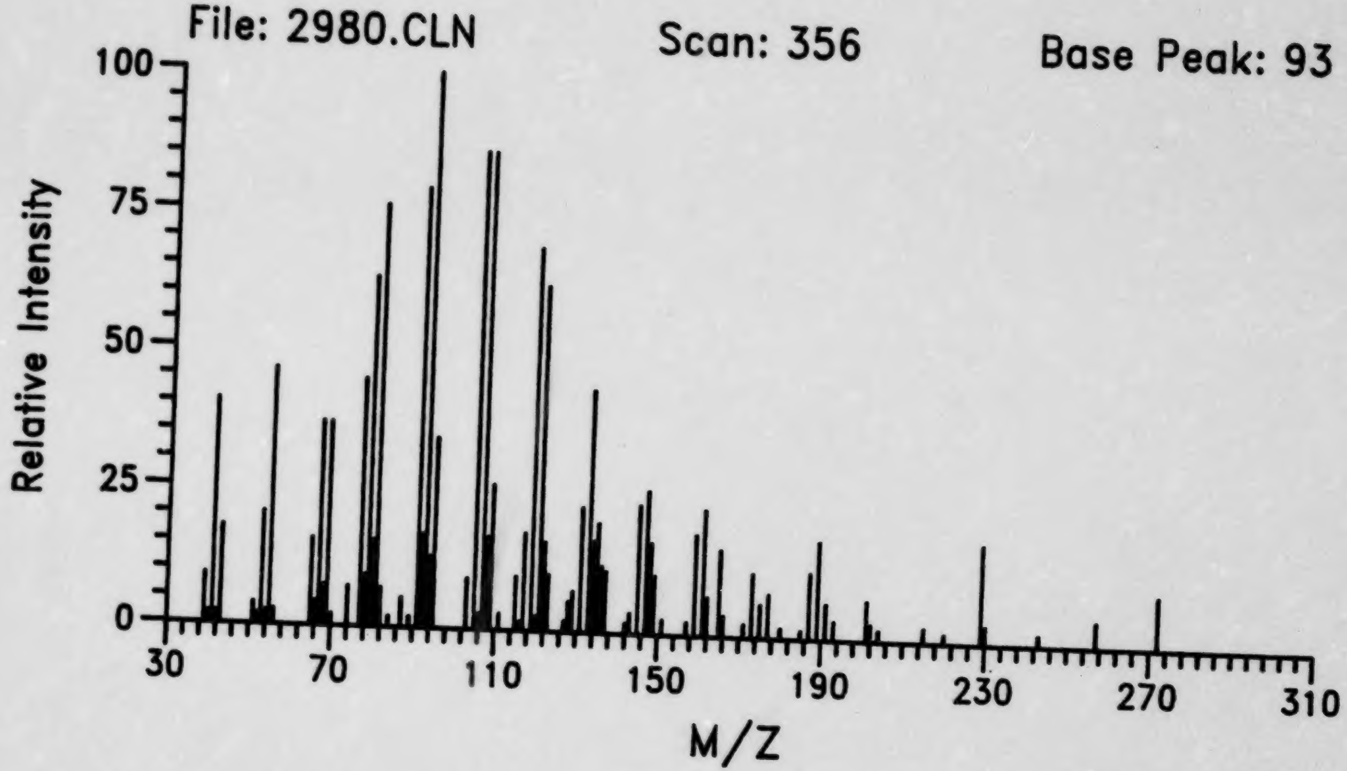


Figure B-26. Extract 2390 (acid), inorganic chemicals industry. Frequency of occurrence: 5.

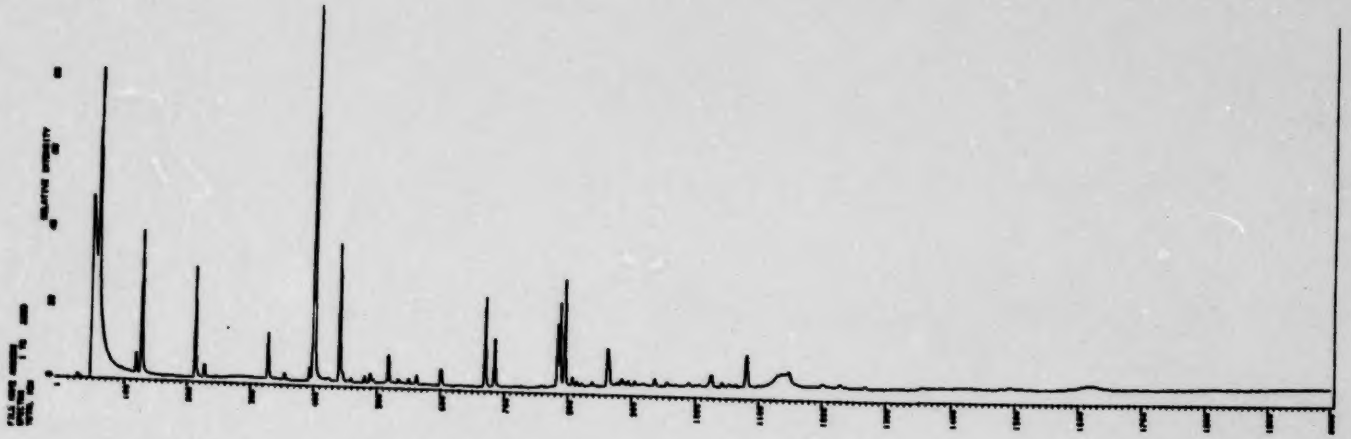


Figure B-27. Capillary chromatogram, extract 2390 (acid), inorganic chemicals industry.

```
RUN IDENTIFICATION 1889 R2390 EPA*13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    758
BACKGROUND    739
MAXIMUM INTENSITY    292         % OF TOTAL ION    10.2
OUTPUT MASS RANGE     40  TO    492
SCAN SPEED      2
MASS           103    102    57   136   138    79    44   115    45    81
INTENSITY    10000   9754  4531  3698  3356  2671  2534  2397  2328  2328
```

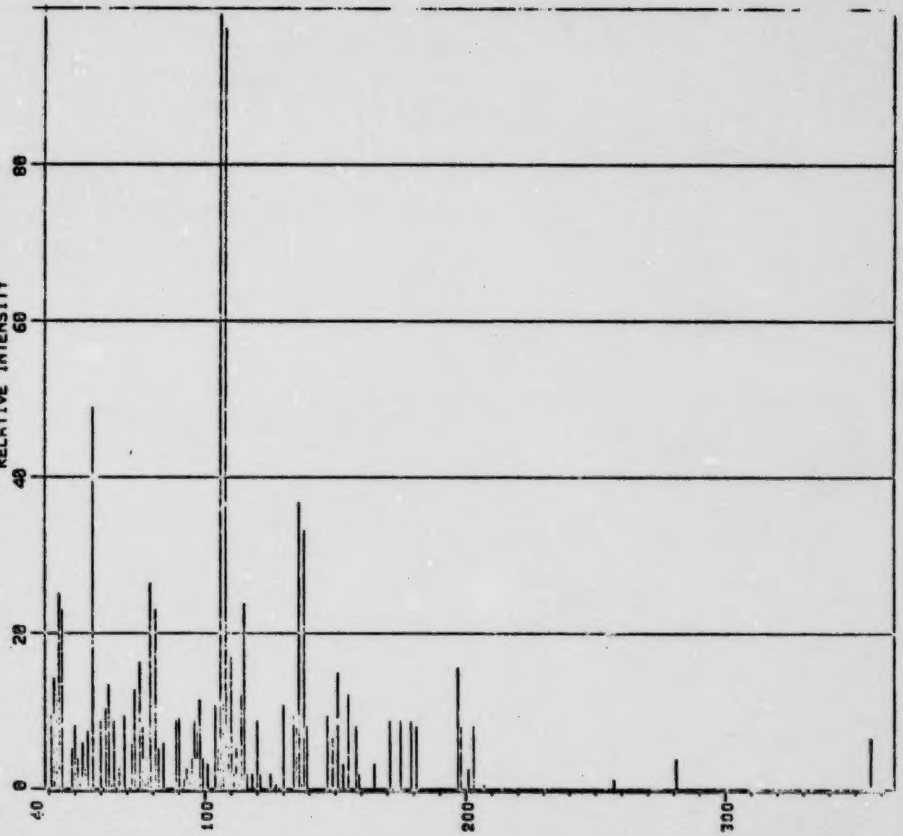
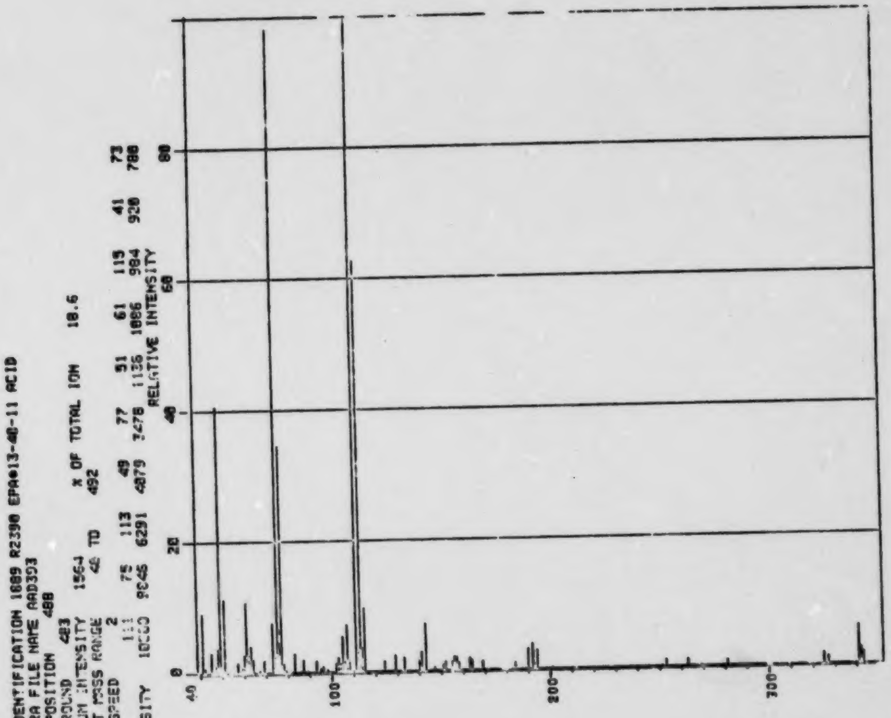


Figure B-28. Mass spectrum, EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, not identified, scan #758.



```
RUN IDENTIFICATION 1089 R2390 EPA●13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    488
BACKGROUND     483
MAXIMUM INTENSITY    1564        % OF TOTAL ION     18.6
OUTPUT MASS RANGE     46  TO    492
SCAN SPEED     2
MASS        111     75    113     49     77     51     61    115     41     73
INTENSITY 10000   9646   6291   4879   3478   1136   1086    984    928    788
```

Figure B-29. Mass spectrum of EPA compound of interest, one possible isomer; extract 2390 (acid), inorganic chemicals industry, scan #488.

```
RUN IDENTIFICATION 1009 R2390 EPA*13-40-11 ACID
SPECTRA FILE NAME AAD333
FILE POSITION   806
BACKGROUND    001
MAXIMUM INTENSITY   1670        % OF TOTAL ION    18.1
OUTPUT MASS RANGE     40 TO    492
SCAN SPEED         2
MASS              113   149    77   115   148    75   150   151    51   114
INTENSITY       10000  3940  3500  3257  2922  2586  2502  2203  1592  1137
```

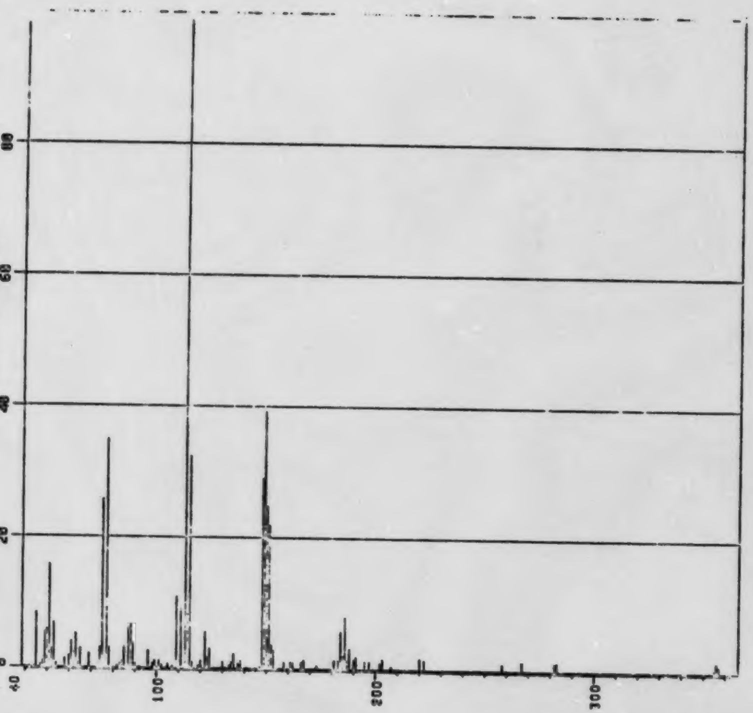


Figure B-30. Mass spectrum of EPA compound of interest, one possible isomer; extract 2390 (acid), inorganic chemicals industry, scan #806.

```
RUN IDENTIFICATION 1689 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION   1147
BACKGROUND   1130
MAXIMUM INTENSITY    752        % OF TOTAL ION    11.6
OUTPUT MASS RANGE     40 TO    492
SCAN SPEED          2
MASS            113   149   151    77    75    51    43   115   184   186
INTENSITY     10000  8324  6429  4654  4335  2351  3111  3085  2234  2074
```

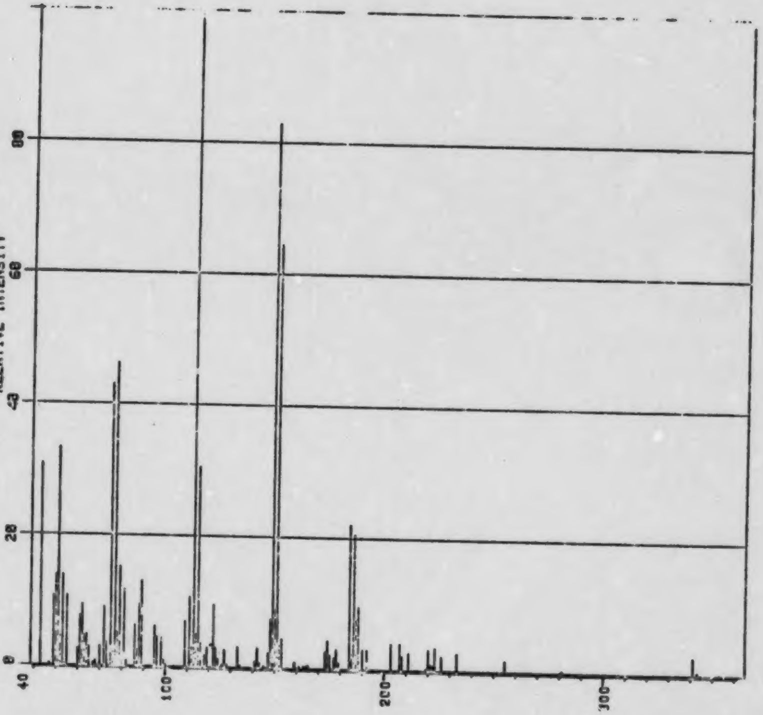
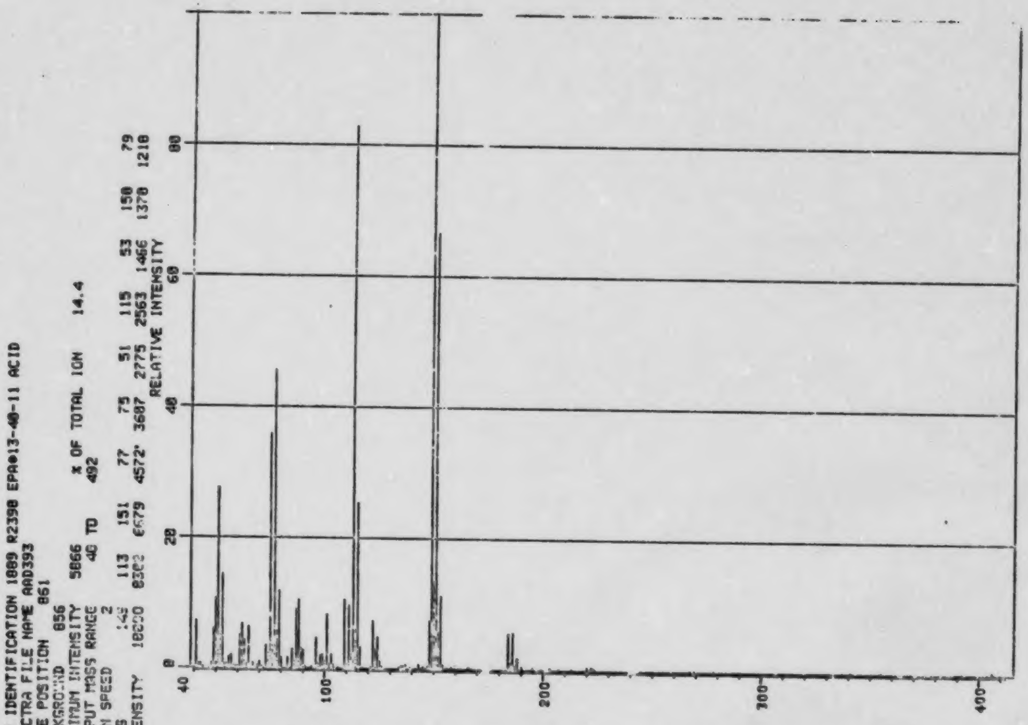


Figure B-31. Mass spectrum of EPA compound of interest, one possible isomer; extract 2390 (acid), inorganic chemicals industry, scan #1147.



```
RUN IDENTIFICATION 1089 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    861
BACKGROUND    856
MAXIMUM INTENSITY    5866         % OF TOTAL ION    14.4
OUTPUT MASS RANGE      40  TO    492
SCAN SPEED         2
MASS          149    113    151     77     75     51    115     53    150     79
INTENSITY   18000   8302   6679  4572'  3687   2775   2563   1466   1370   1218
```

Figure B-32. Mass spectrum, EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #861.

```
RUN IDENTIFICATION 1089 R2390 EPA*13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION   1079
BACKGROUND   1056
MAXIMUM INTENSITY   4364      % OF TOTAL ION     14.3
OUTPUT MASS RANGE    40 TO    492
SCAN SPEED     2
MASS          145   151   113    77    75    51   115   181   189   184
INTENSITY   18260  6769  6296  4592  4248  2447  1714  1590  1507  1278
```

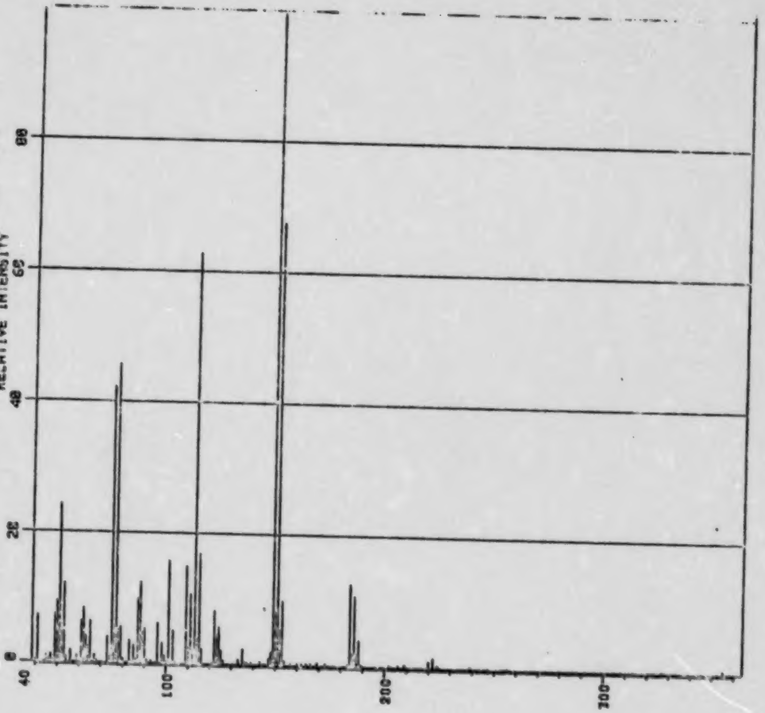


Figure B-33. Mass spectrum, EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #1079.

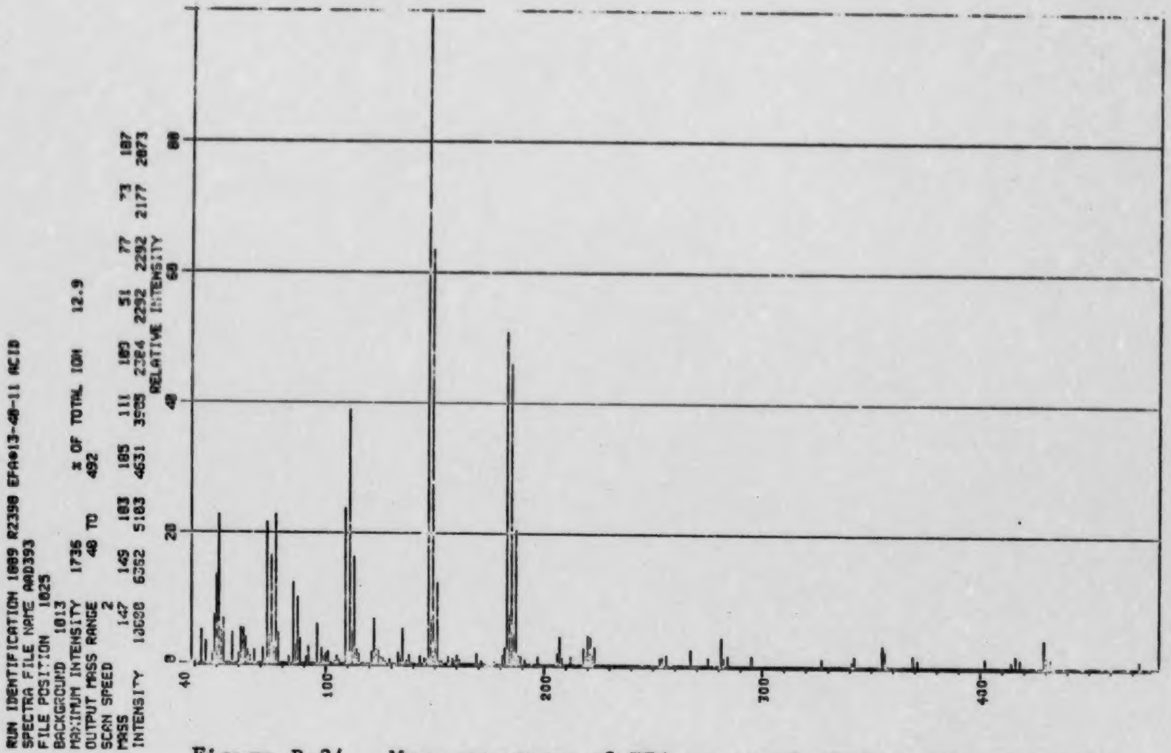
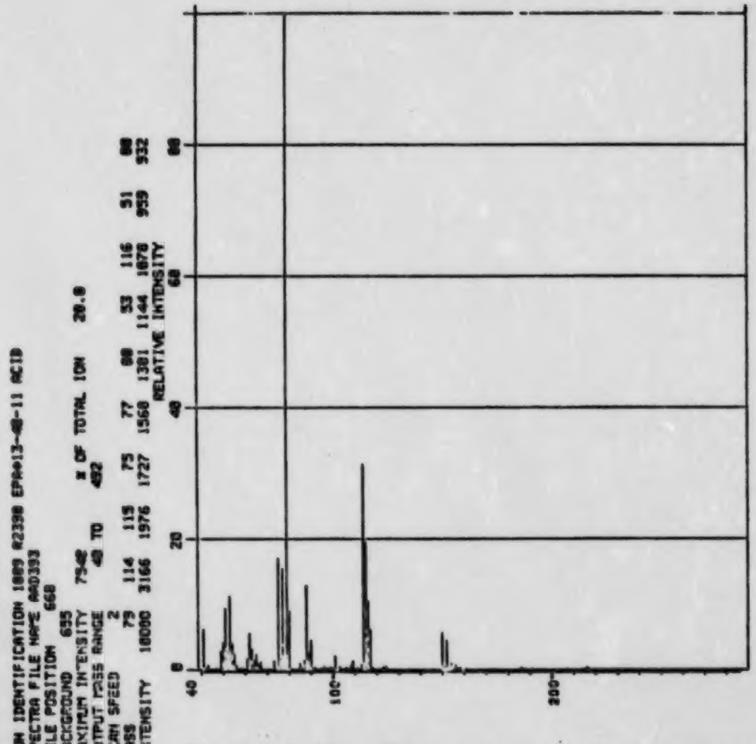


Figure B-34. Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, scan #1025.



```
RUN IDENTIFICATION 1889 R2390 EPA*13-48-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION     668
BACKGROUND      655
MAXIMUM INTENSITY     7542               % OF TOTAL ION     20.8
OUTPUT MASS RANGE      40  TO   492
SCAN SPEED        2
MASS           79    114    115    75    77    80    53    116    51    80
INTENSITY   10000   3166   1976  1727  1568  1381  1144   1078   959   932
```

Figure B-35. Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #668.

```
RUN IDENTIFICATION 1009 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AA2393
FILE POSITION    684
BACKGROUND     655
MAXIMUM INTENSITY  15744        % OF TOTAL ION    30.0
OUTPUT MASS RANGE     40 TO     492
SCAN SPEED         2
MASS          115    79   117    75    77    41    91    53    65   116
INTENSITY   10000  3766  3370  1427  1347   974   926   895   769   764
```

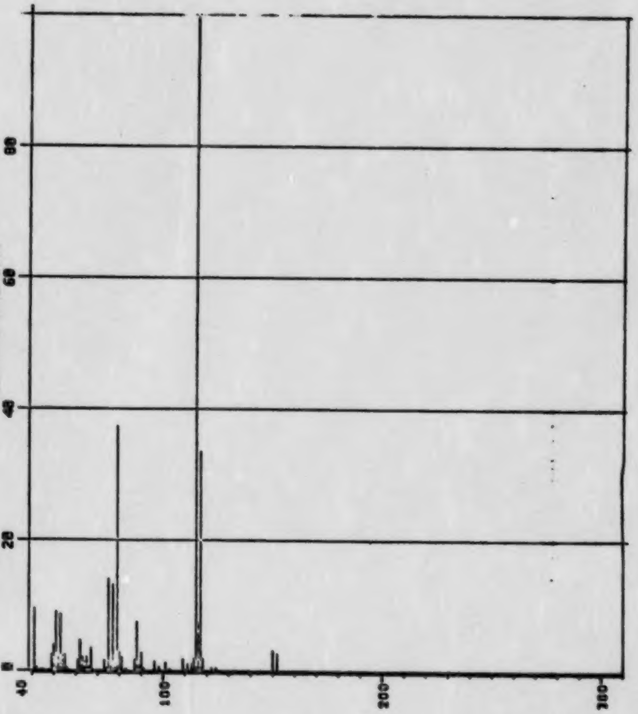
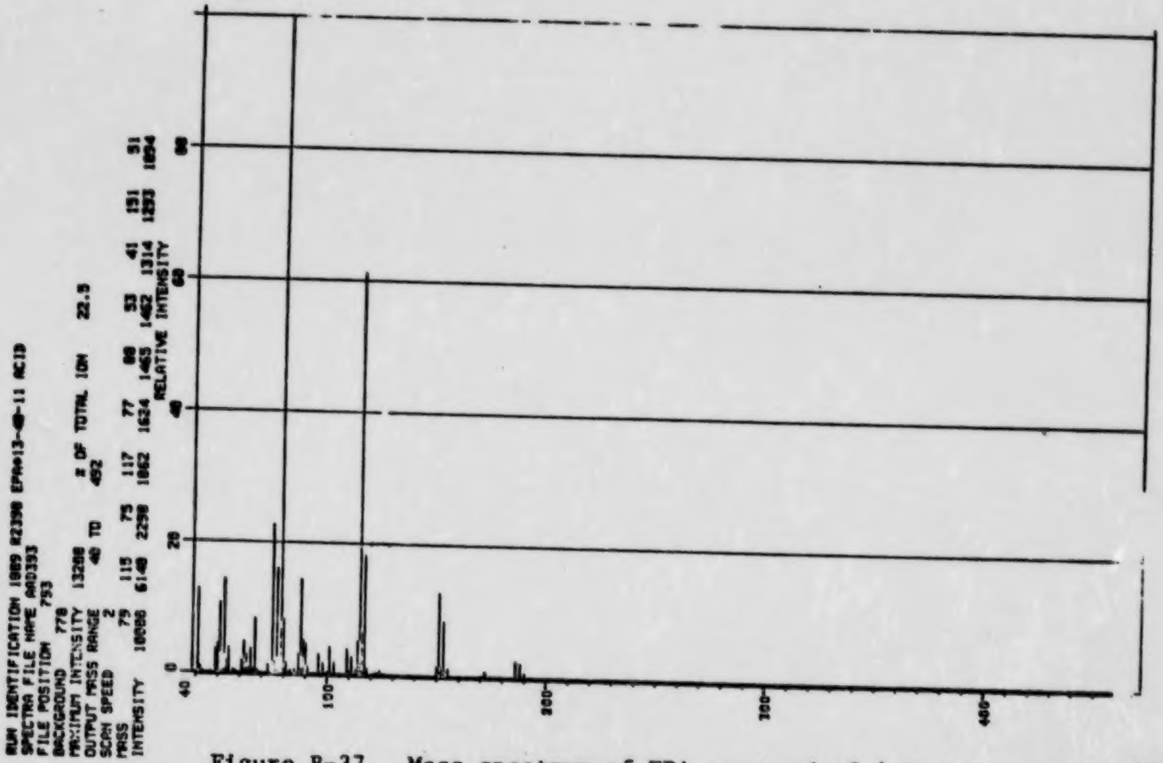
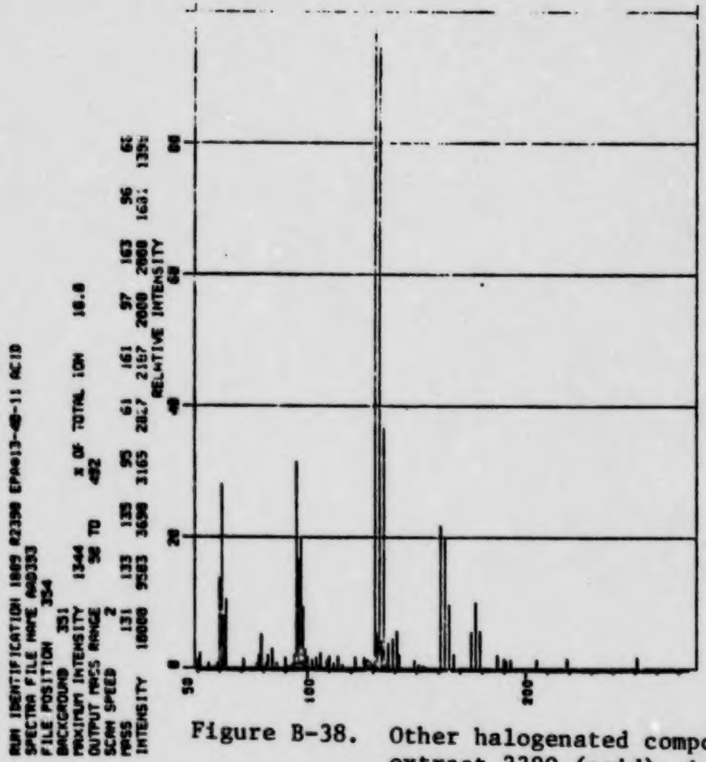


Figure B-36. Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #684.



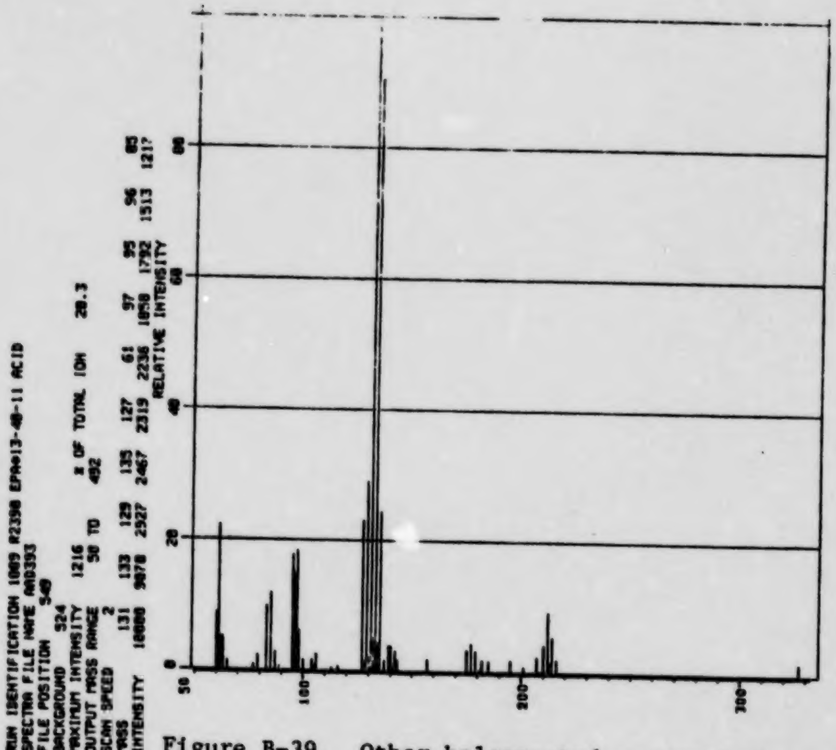
```
RUN IDENTIFICATION 1889 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION     793
BACKGROUND      778
MAXIMUM INTENSITY  13208        % OF TOTAL ION    22.5
OUTPUT MASS RANGE    40  TO    492
SCAN SPEED        2
MASS          79    115    75    117    77    88    53    41    131    51
INTENSITY   10000  6140  2298  1862  1624  1465  1462  1314  1293  1054
```

Figure B-37. Mass spectrum of EPA compound of interest, extract 2390 (acid), inorganic chemicals industry, one isomer, scan #793.



```
RUN IDENTIFICATION 1809 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AA0353
FILE POSITION    354
BACKGROUND    351
MAXIMUM INTENSITY   1344        % OF TOTAL ION    18.8
OUTPUT MASS RANGE     50 TO    492
SCAN SPEED       2
MASS             131   133   135    95    61   161    97   163    96    63
INTENSITY      10000  9583  3690  3165  2827  2167  2000  2000  1631  1395
```

Figure B-38. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #354.



```
RUN IDENTIFICATION 1009 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    549
BACKGROUND       524
MAXIMUM INTENSITY     1216        % OF TOTAL ION    28.3
OUTPUT MASS RANGE      50 TO    492
SCAN SPEED          2
MASS          131    133    129    135    127     61     97     95     96     85
INTENSITY   10000   9878   2927   2467   2319   2236   1858   1792   1513   1217
```

Figure B-39. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #549.

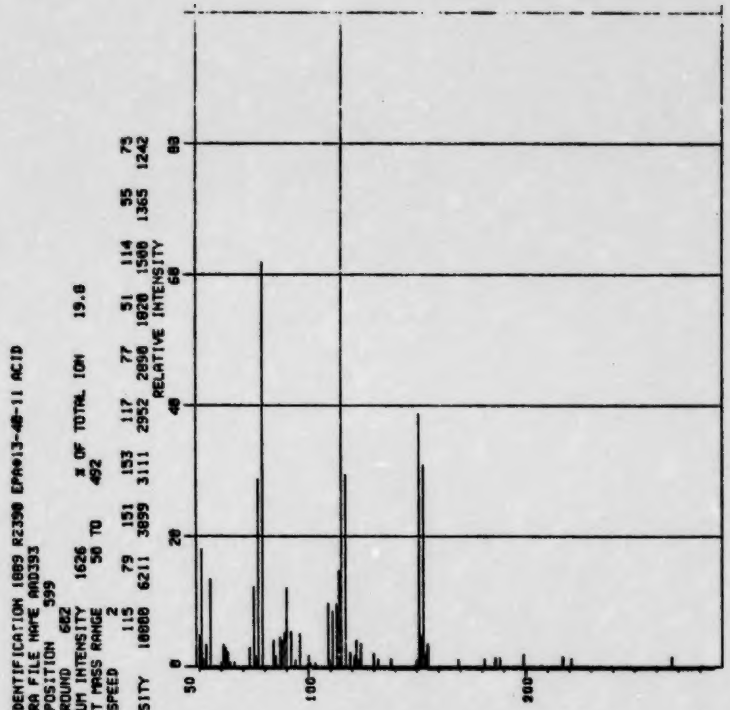


Figure B-40. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #599.

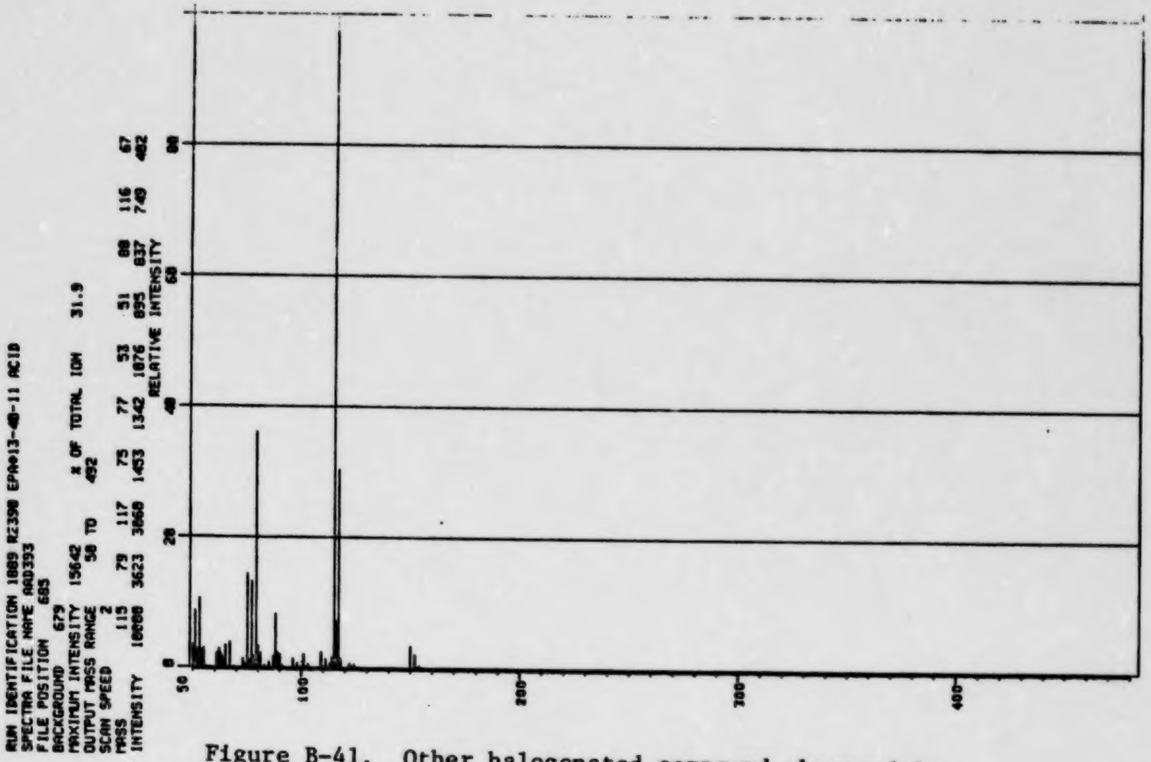


Figure B-41. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #685.

```
RUN IDENTIFICATION 1089 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AR0393
FILE POSITION    794
BACKGROUND     791
MAXIMUM INTENSITY  27092        % OF TOTAL ION    24.0
OUTPUT MASS RANGE     40 TO    492
SCAN SPEED        2
MASS          79   115    75   117    41    77    53    80   151    91
INTENSITY  10000  5789  2456  1843  1560  1545  1317  1251  1133  1015
```

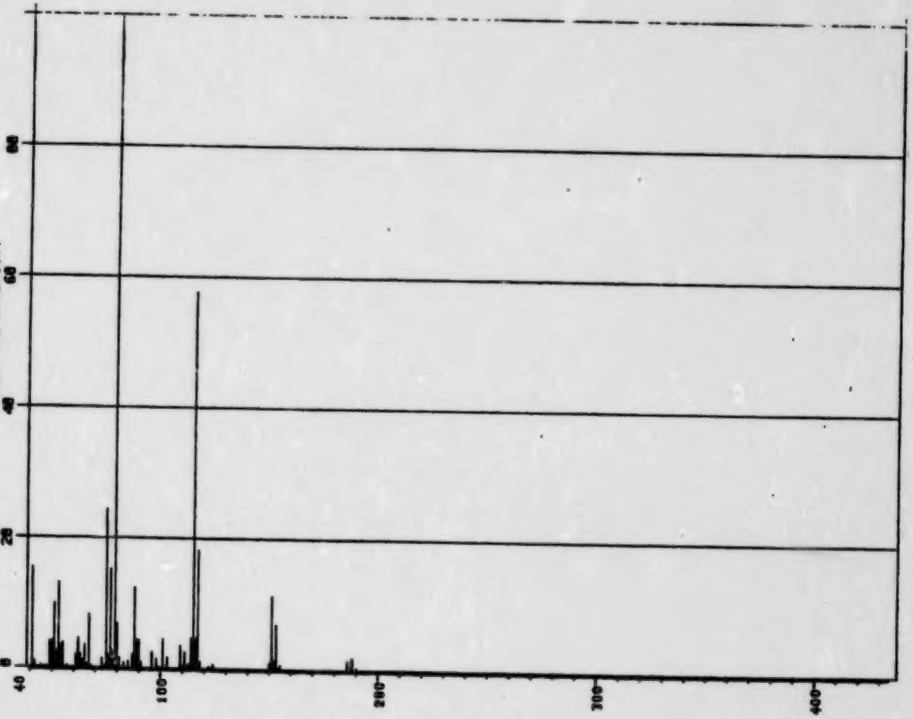


Figure B-42. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #794.

```
RUN IDENTIFICATION 1089 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME ARD393
FILE POSITION    806
BACKGROUND     837
MAXIMUM INTENSITY    1664       % OF TOTAL ION    19.5
OUTPUT MASS RANGE     40  TO   492
SCAN SPEED        2
MASS          113   149   115    77    75   148   150   151    51   189
INTENSITY   10000  3882  3846  3569  3020  2900  2355  2211  1574  1141
```

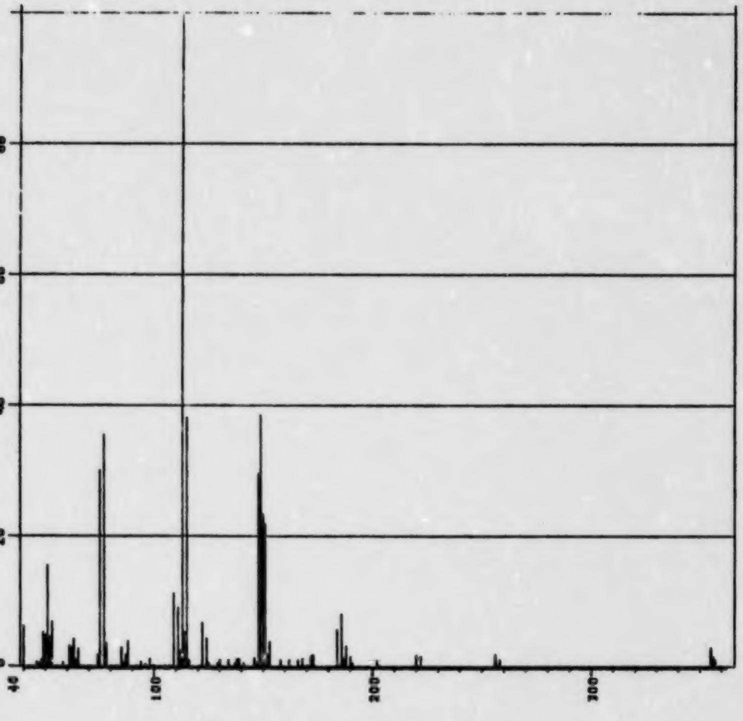
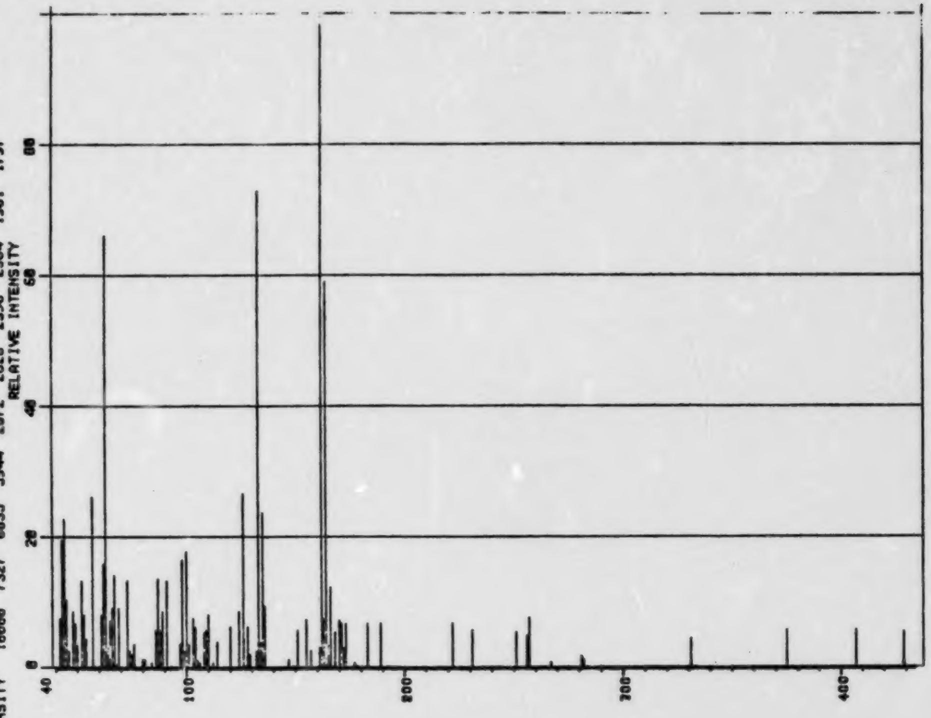
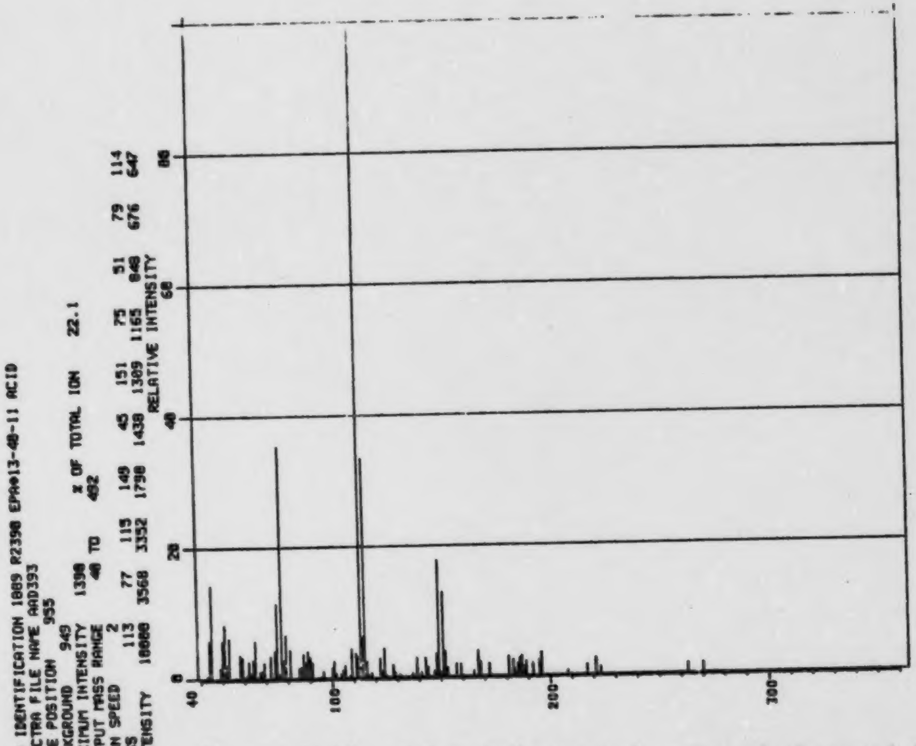


Figure B-43. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #806.



```
RUN IDENTIFICATION 1889 R2390 EPA*13-48-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    877
BACKGROUND     871
MAXIMUM INTENSITY      434            % OF TOTAL ION     11.8
OUTPUT MASS RANGE       48  TO   492
SCAN SPEED          2
MASS          162    133     63    164    126     57    135     44     43    188
INTENSITY   10000   7327   6635   5944   2672   2626   2396   2384   1981   1797
```

Figure B-44. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #877.



```
RUN IDENTIFICATION 1009 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    955
BACKGROUND      949
MAXIMUM INTENSITY    1390        % OF TOTAL ION    22.1
OUTPUT MASS RANGE     40   TO    492
SCAN SPEED        2
MASS          113    77   115   149    45   151    75    51    79   114
INTENSITY   10000  3560  3352  1790  1430  1309  1165   840   676   647
```

Figure B-45. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #955.

```
RUN IDENTIFICATION 1889 R2390 EPA*13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    1005
BACKGROUND    1000
MAXIMUM INTENSITY      352      % OF TOTAL ION    16.4
OUTPUT MASS RANGE      40 TO    492
SCAN SPEED      2
MASS          113     44    149    115    151     77     45    135     74    150
INTENSITY   10000   3636   3060   2500   2329   2272   2215   1363   1250   1250
```

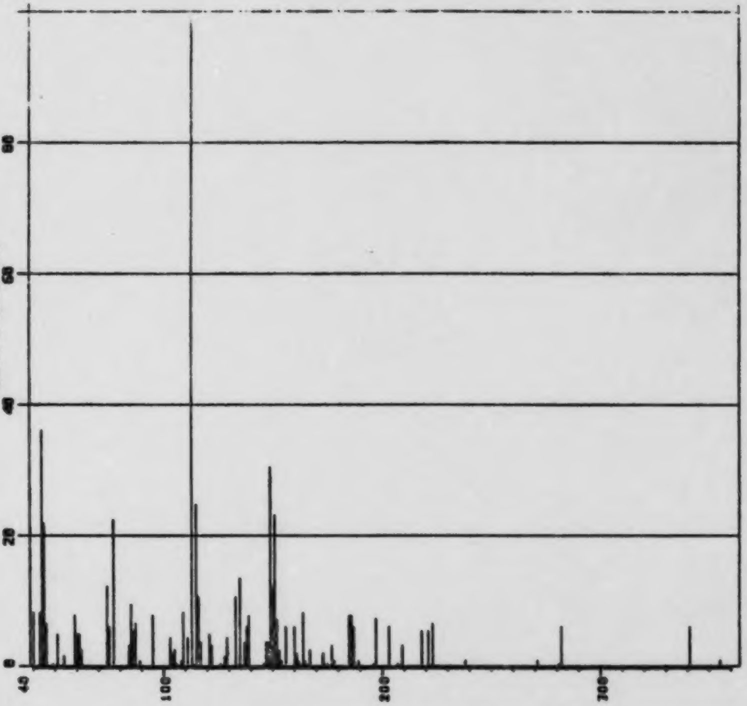


Figure B-46. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1005.

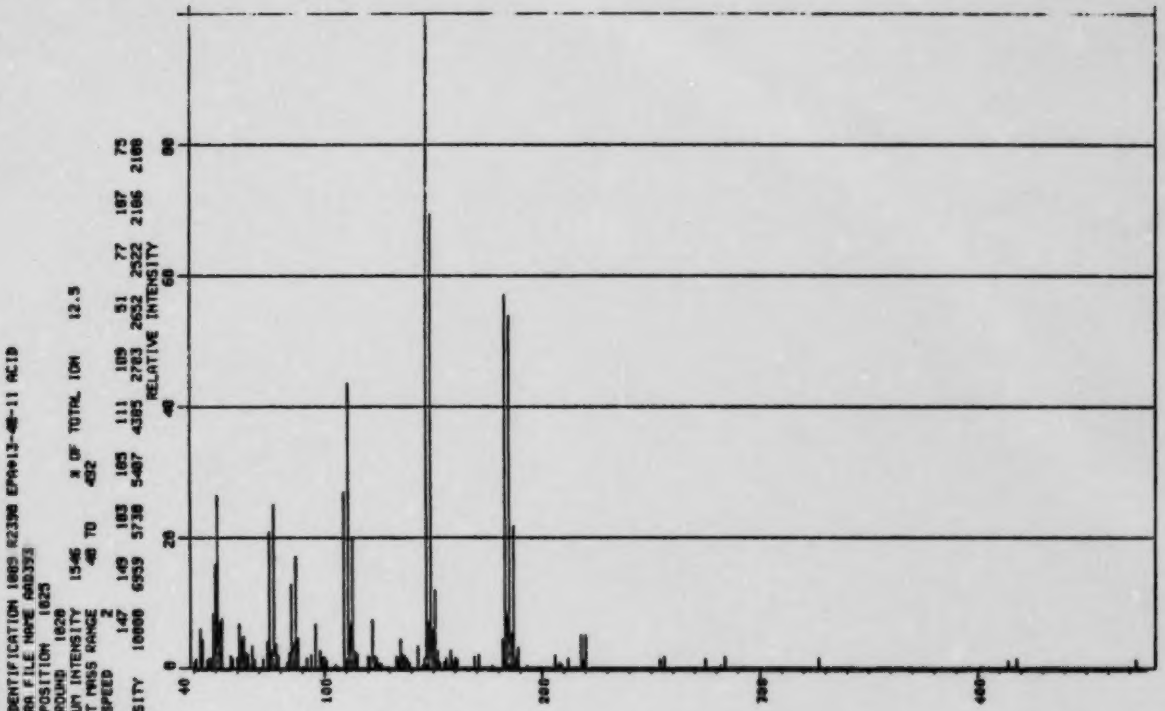
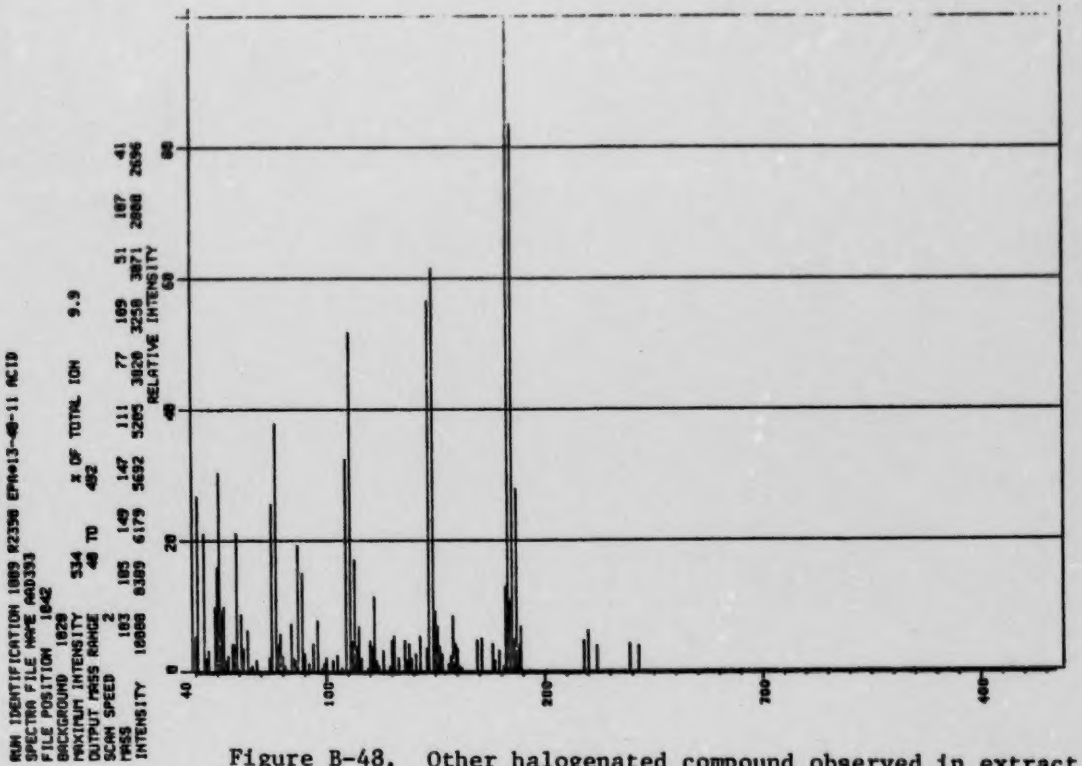


Figure B-47. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1025.



```
RUN IDENTIFICATION 1009 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD393
FILE POSITION    1042
BACKGROUND    1020
MAXIMUM INTENSITY     534          % OF TOTAL ION     9.9
OUTPUT MASS RANGE      40 TO     492
SCAN SPEED          2
MASS              183    185    149    147    111     77    109     51    107     41
INTENSITY       10000   8389   6179   5692   5205   3820   3250   3071   2800   2696
```

Figure B-48. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1042.

```
RUN IDENTIFICATION 1089 R2390 EPA#13-40-11 ACID
SPECTRA FILE NAME AAD392
FILE POSITION   1080
BACKGROUND    1070
MAXIMUM INTENSITY    4956        % OF TOTAL ION    14.4
OUTPUT MASS RANGE     40 TO    452
SCAN SPEED       2
MASS         149    151    113     75     77     51    115     53    101    109
INTENSITY  10000   6100   5040   4767   3907   2301   2073   1601   1641   1377
```

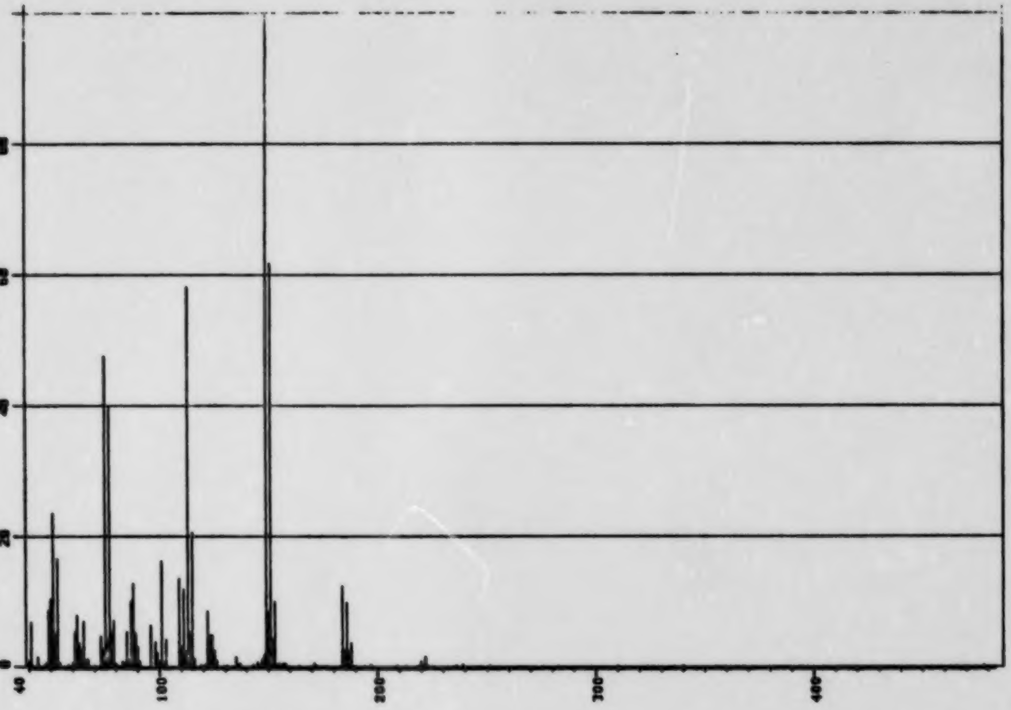


Figure B-49. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1080.

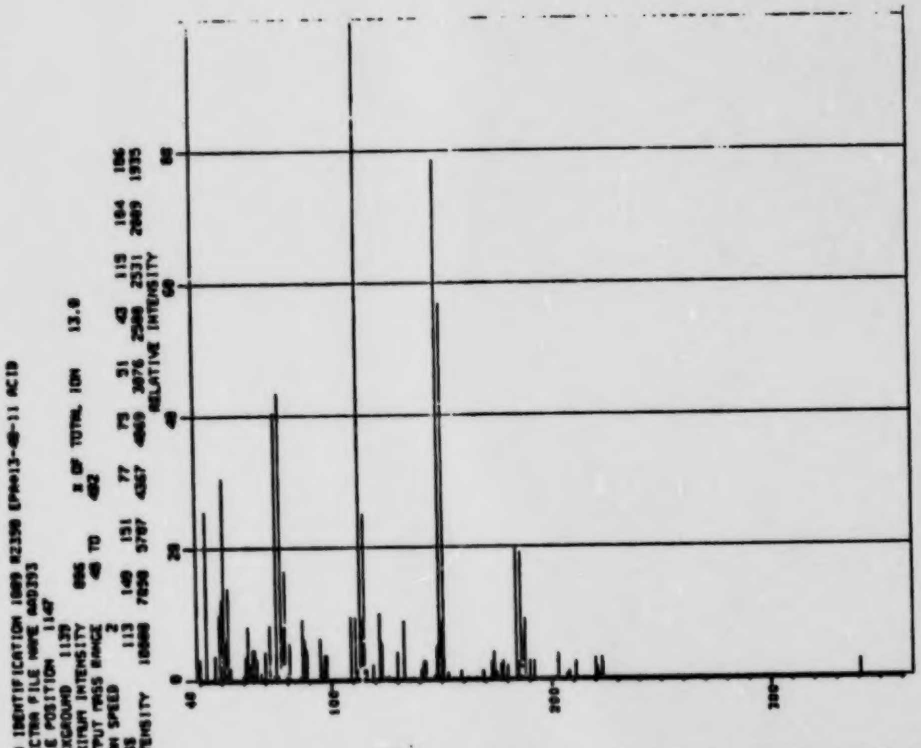


Figure B-50. Other halogenated compound observed in extract 2390 (acid), inorganic chemicals industry, spectrum unidentified, scan #1147.

Figure B-51. Table of Compounds Identified and Tentatively Identified

Scan	Compound
57	chloroform
128	bromodichloromethane
212	1,1,2,2-tetrachloroethane
328	dibromodichloroethane
393	1,2,3-trichloropropane
439	1,1,2,2-tetrachloroethane
489	bromodichloropropane isomer
492	bromodichloropropane isomer
861	trichlorocyclohexene isomer
882	trichlorocyclohexane isomer

Figure B-52. Conclusions

This extract contains an abundance of chlorinated and chlorinated/brominated alkanes, alkenes, cyclopentanes, cyclohexanes, and cyclohexenes. Some of the compounds can be identified, but most are recognizable only as halogenated organics. Chemical ionization mass spectrometry adds data but no real information. Standards for polyhalo alkanes, alkenes, and cyclic hydrocarbons are not available.

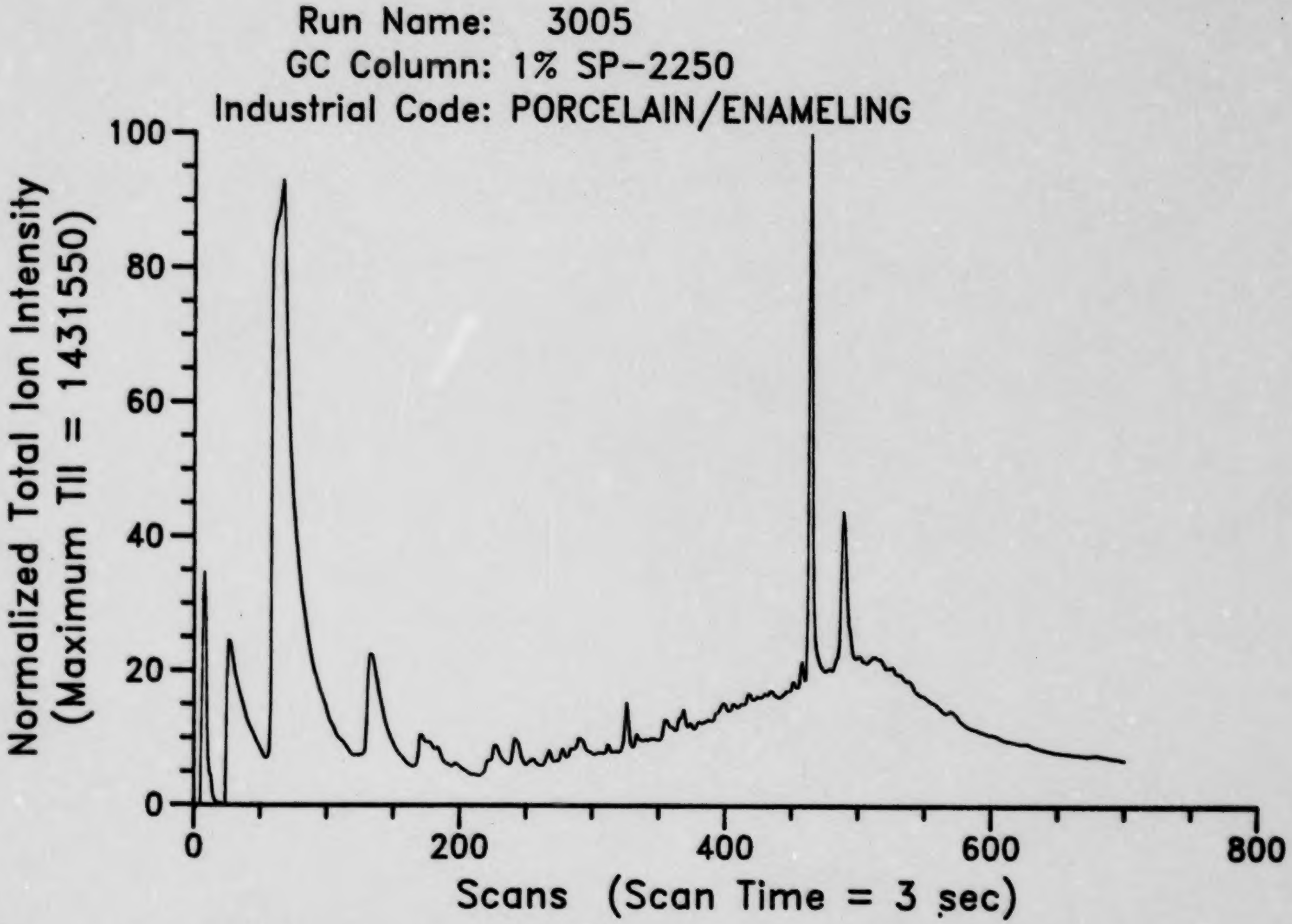


Figure B-53. Packed column chromatogram, extract 3005 (base/neutral), porcelain/enameling industry.

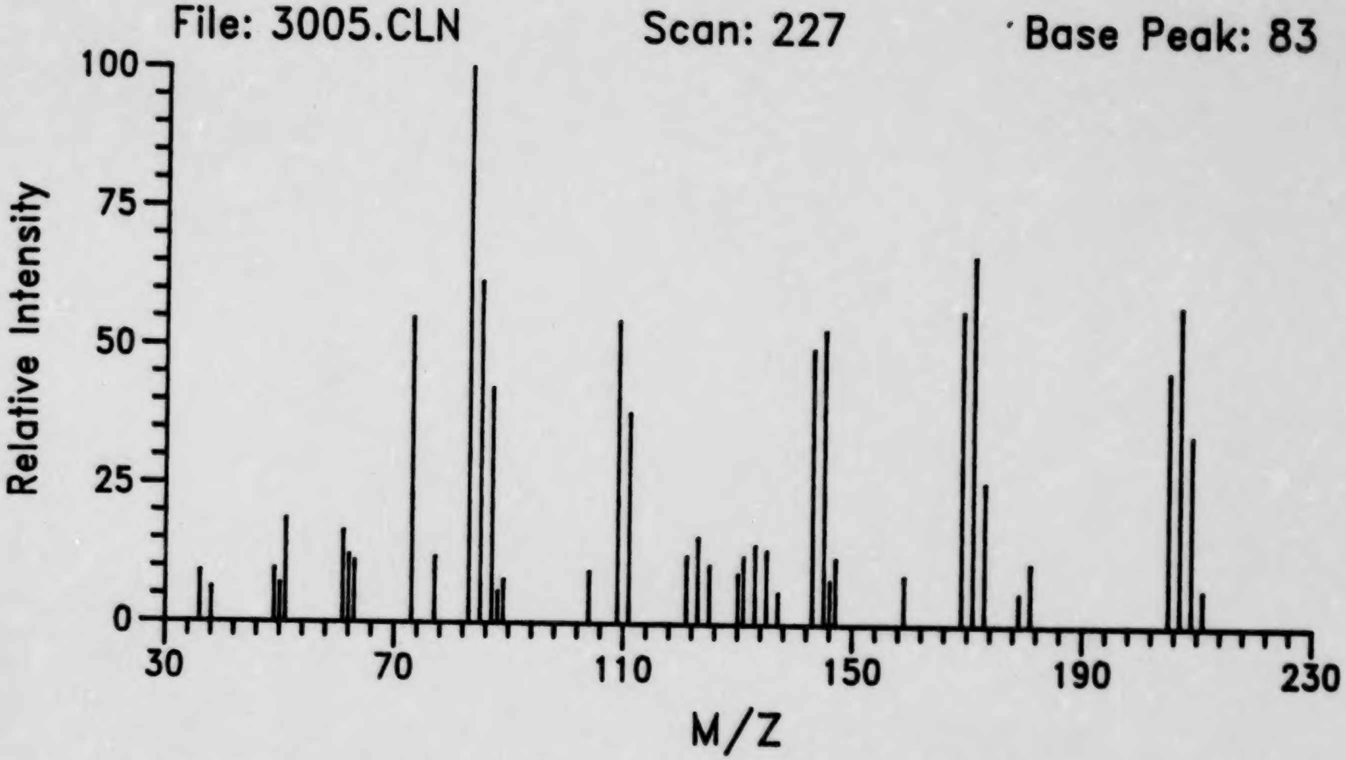


Figure B-54. Extract 3005 (base/neutral), porcelain/enameling industry.
Frequency of occurrence: 27.

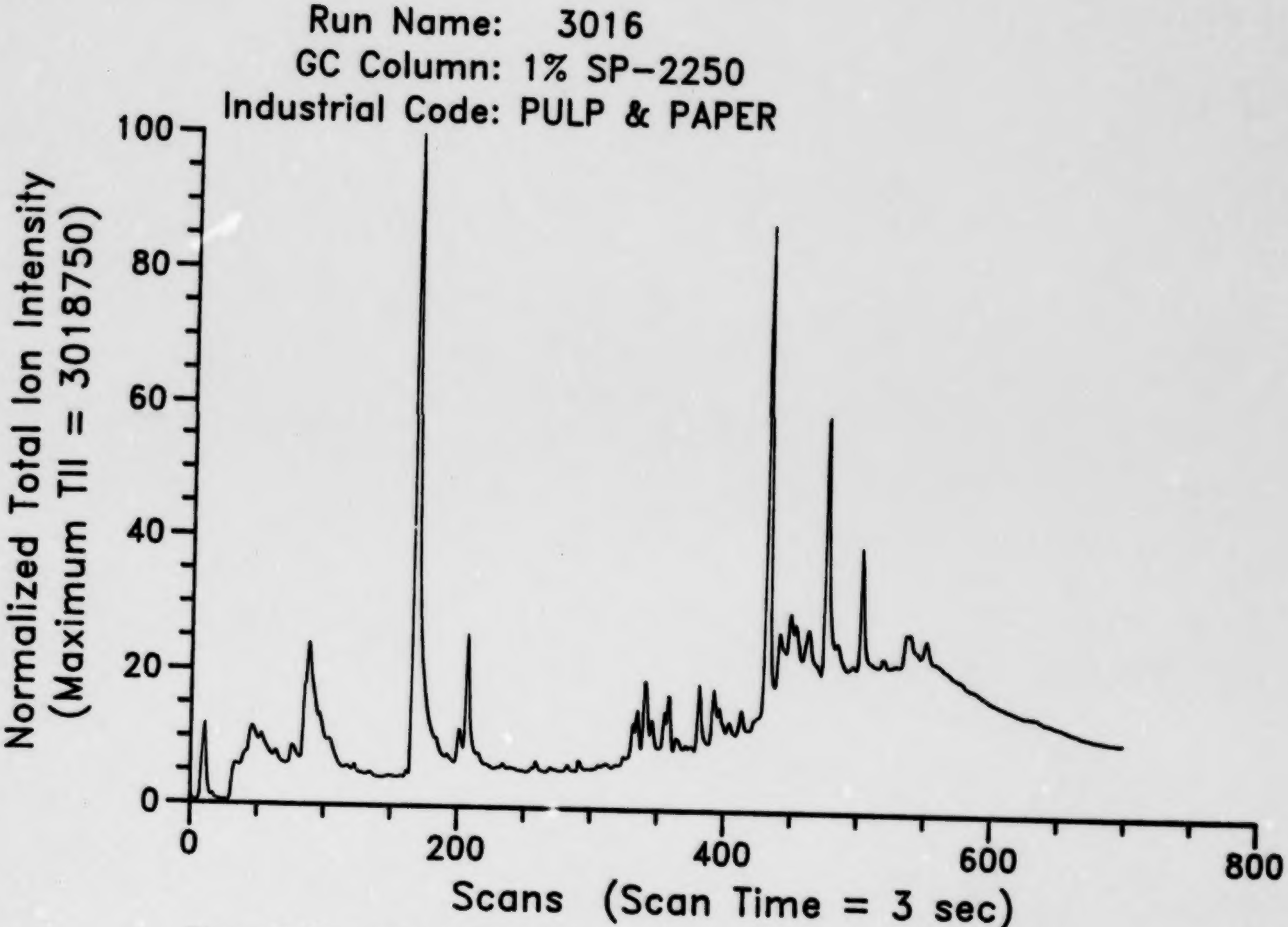


Figure B-55. Packed column chromatogram of extract 3016 (base/neutral), pulp and paper industry. (NOTE: This extract was actually supplied and analyzed rather than extract 3006 (base/neutral).

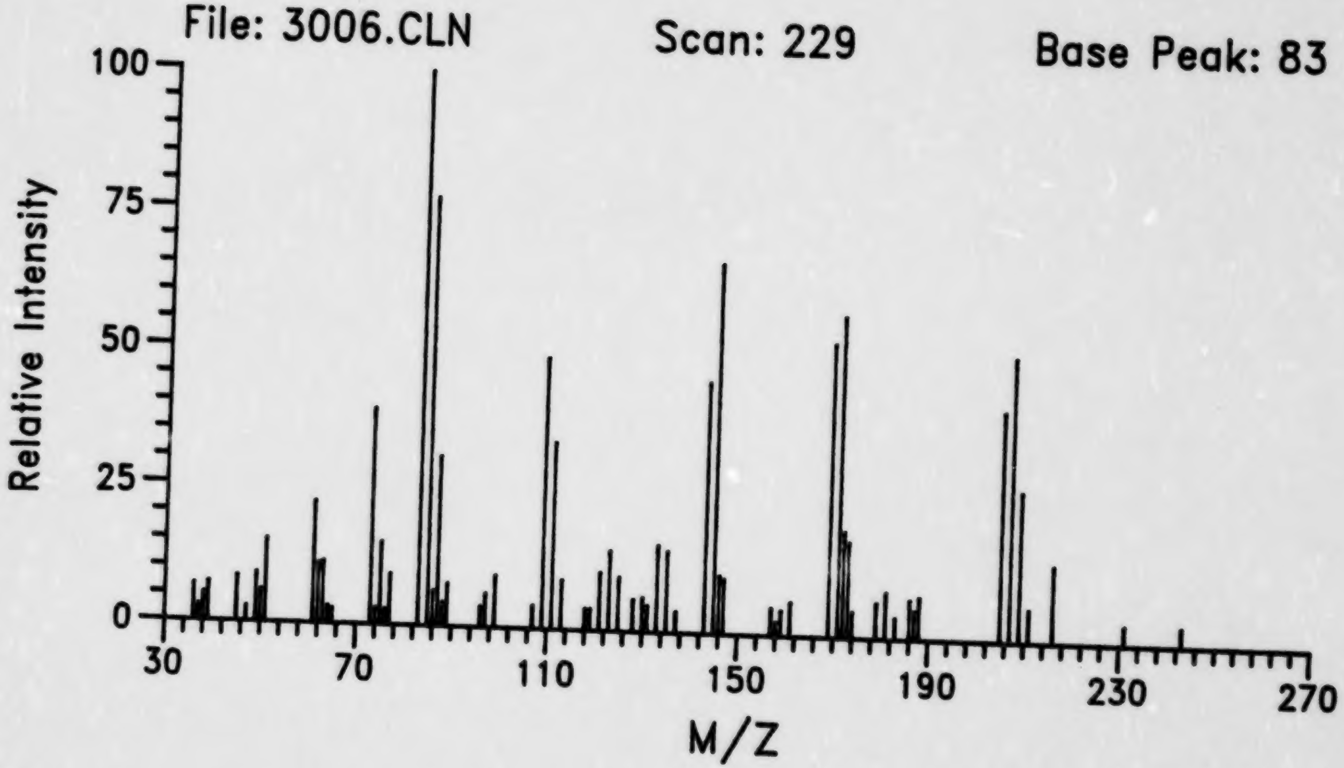


Figure B-56. Extract 3006 (base/neutral), porcelain/enameling industry. Frequency of occurrence: 27.

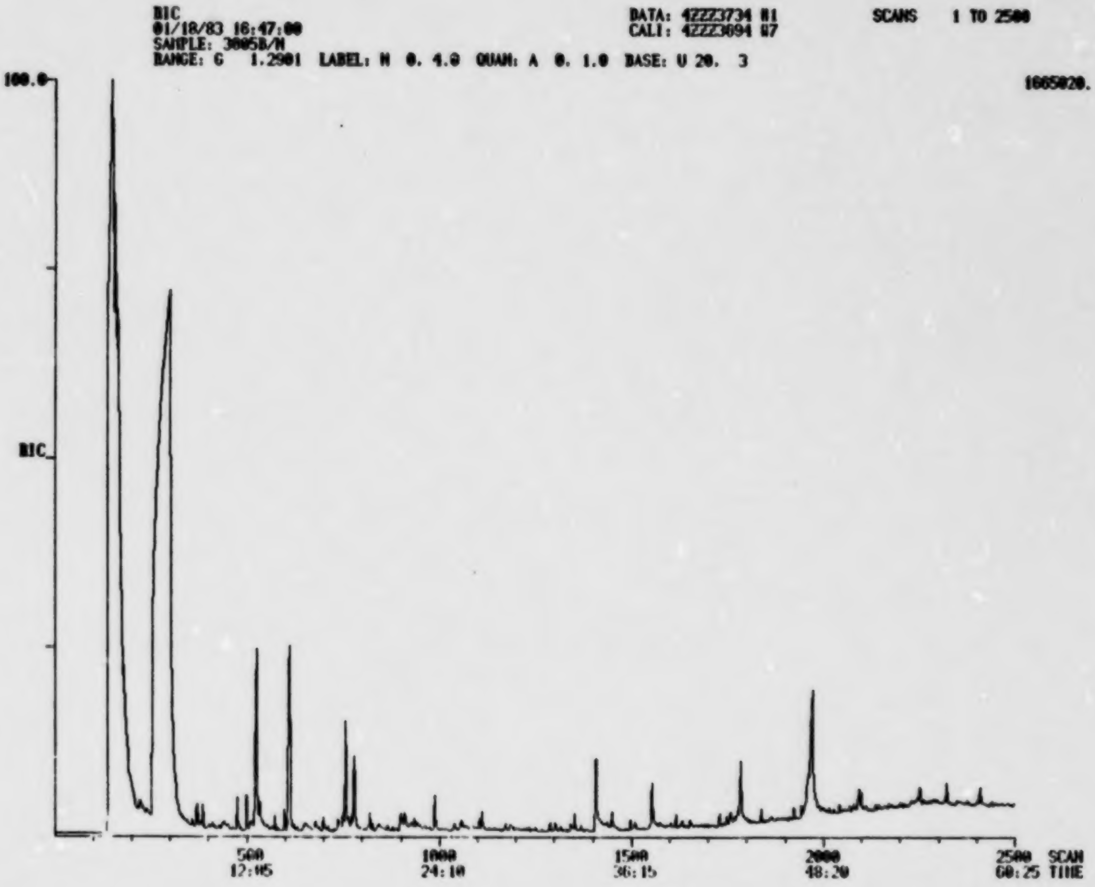


Figure B-57. Capillary chromatogram, extract 3005 (base/neutral), porcelain/enameling industry.

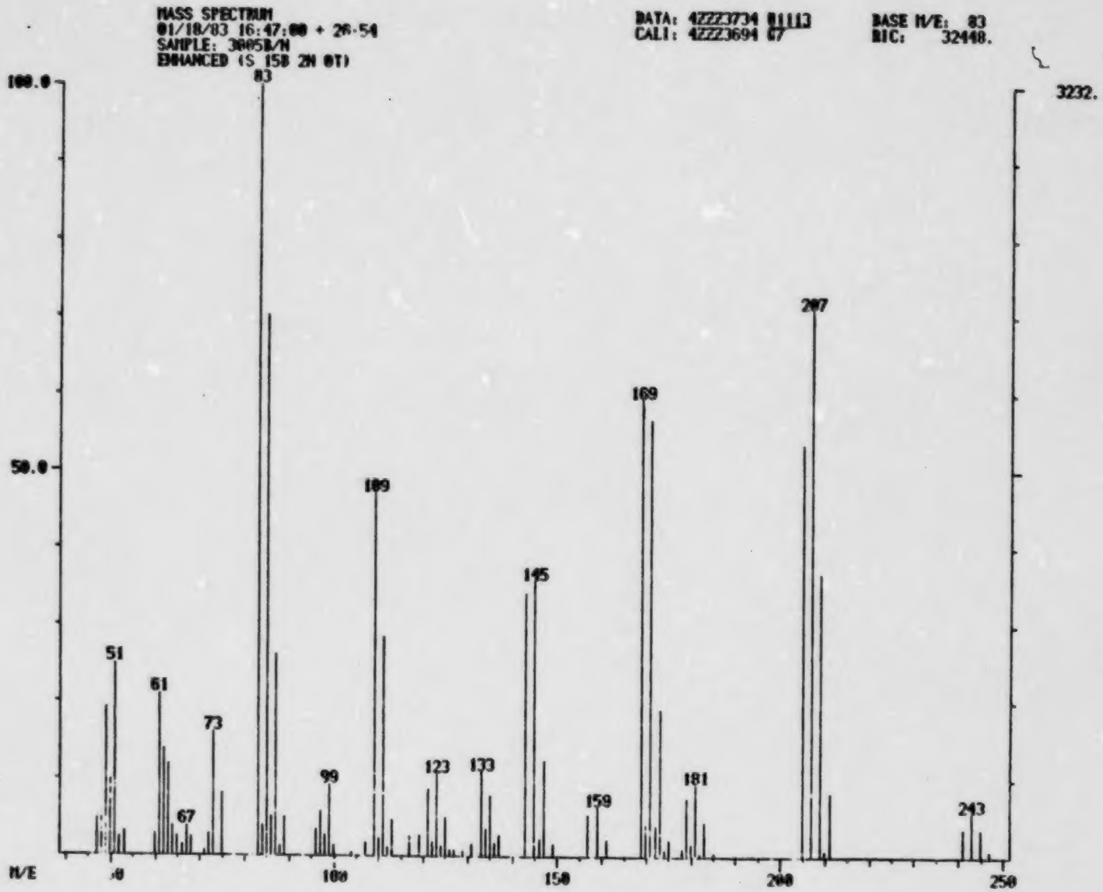


Figure B-58. EPA compound of interest, extract 3005 (base/neutral), porcelain/enameling industry, scan #1113.

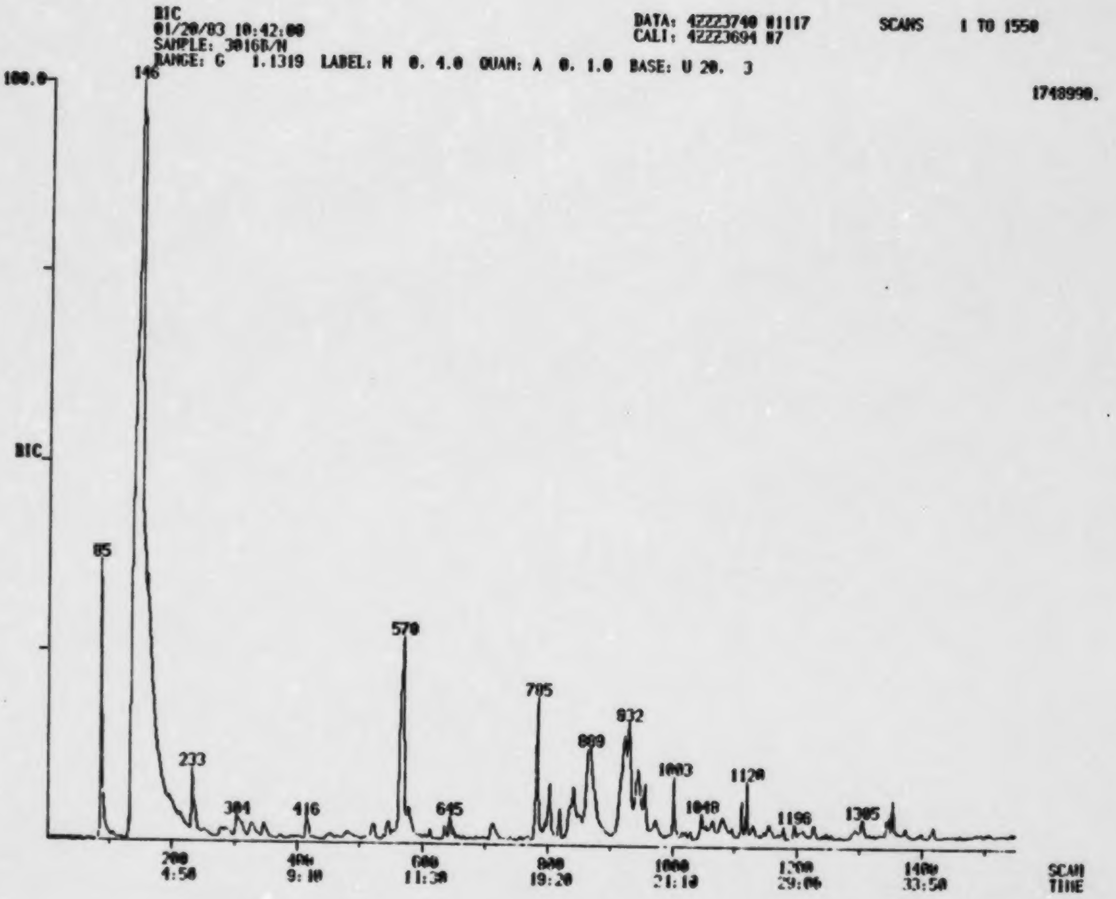


Figure B-59. Capillary chromatogram, extract 3016 (base/neutral), pulp and paper industry, scan #1117.

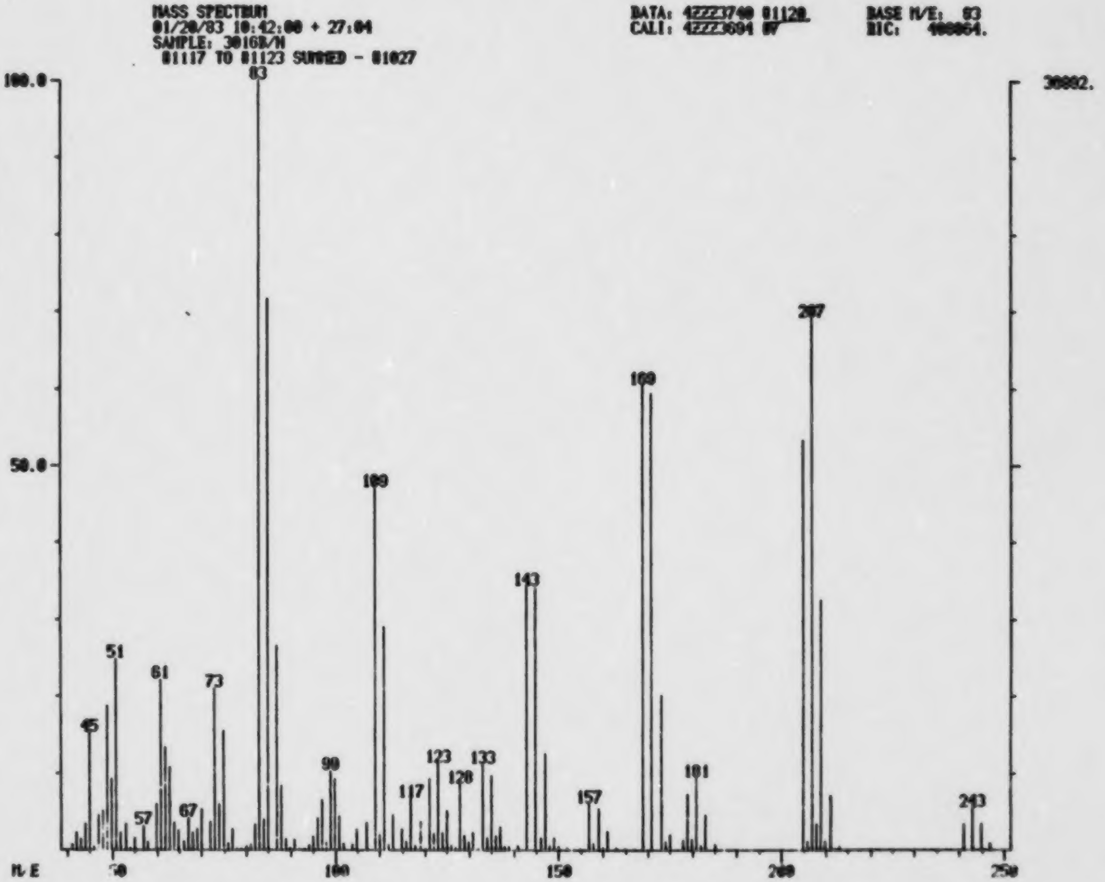


Figure B-60. Mass spectrum, EPA compound of interest, extract 3016 (base/neutral), pulp and paper industry, scan #1120.

Figure B-61. Conclusions

Accurate mass measurement, consistent with the profile of the halogen cluster at m/z 241, suggests a composition of $C_5H_6Cl_5$ (error, 1.4 mmu) for this ion. The ion is almost surely a fragment ion, but no ion of higher mass is observed using chemical ionization techniques. If the number of carbon atoms is correct, this ion could represent only a loss of Cl from a molecular ion--i.e., a saturated C_5 species can only be C_5X_{12}. This information would mean that the compound is a hexachloropentane, a family for which no standard spectra are present in compendia and no standards are commercially available.

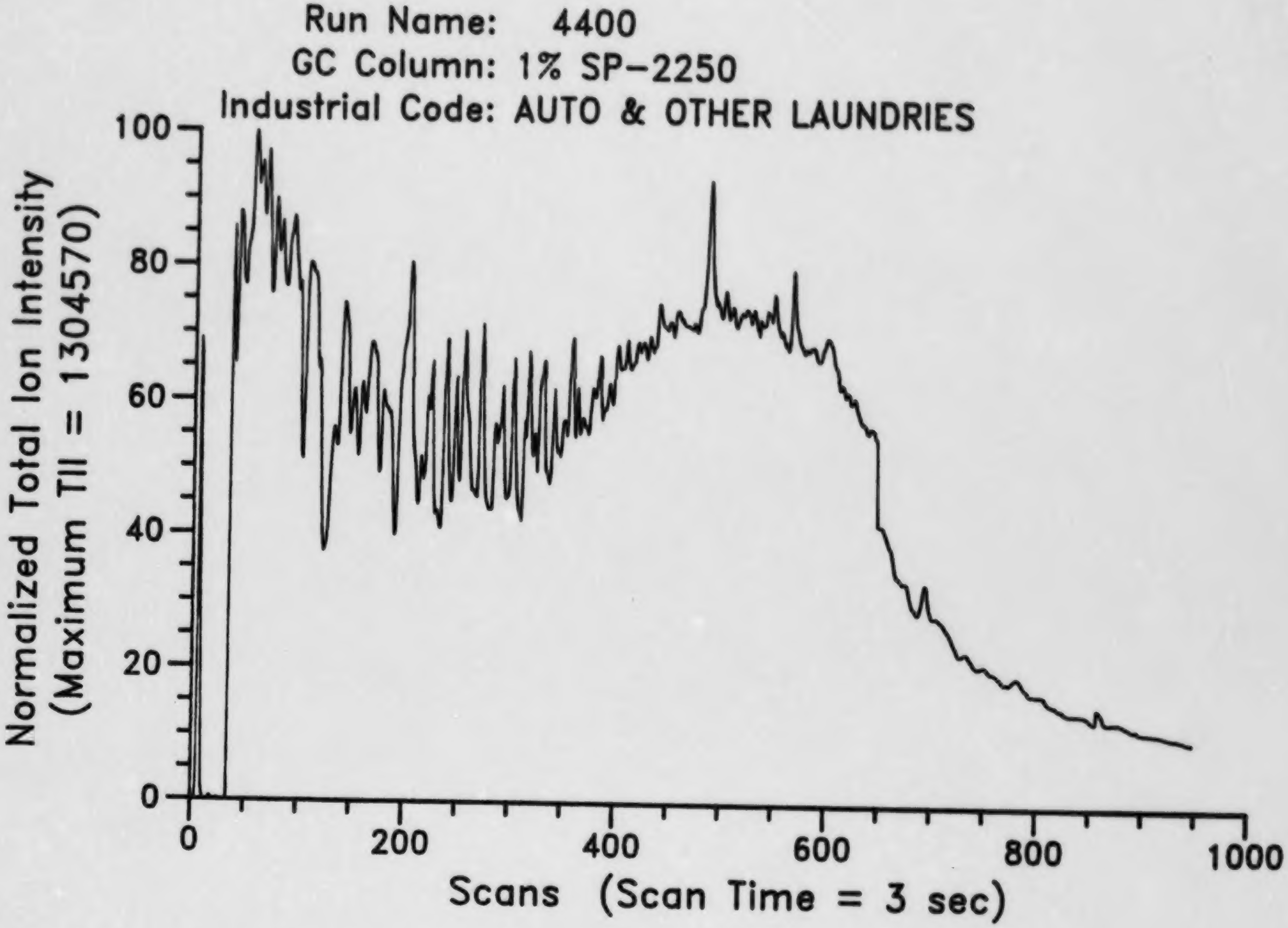


Figure B-62. Packed column chromatogram, extract 4400 (base/neutral), auto and other laundries industry.

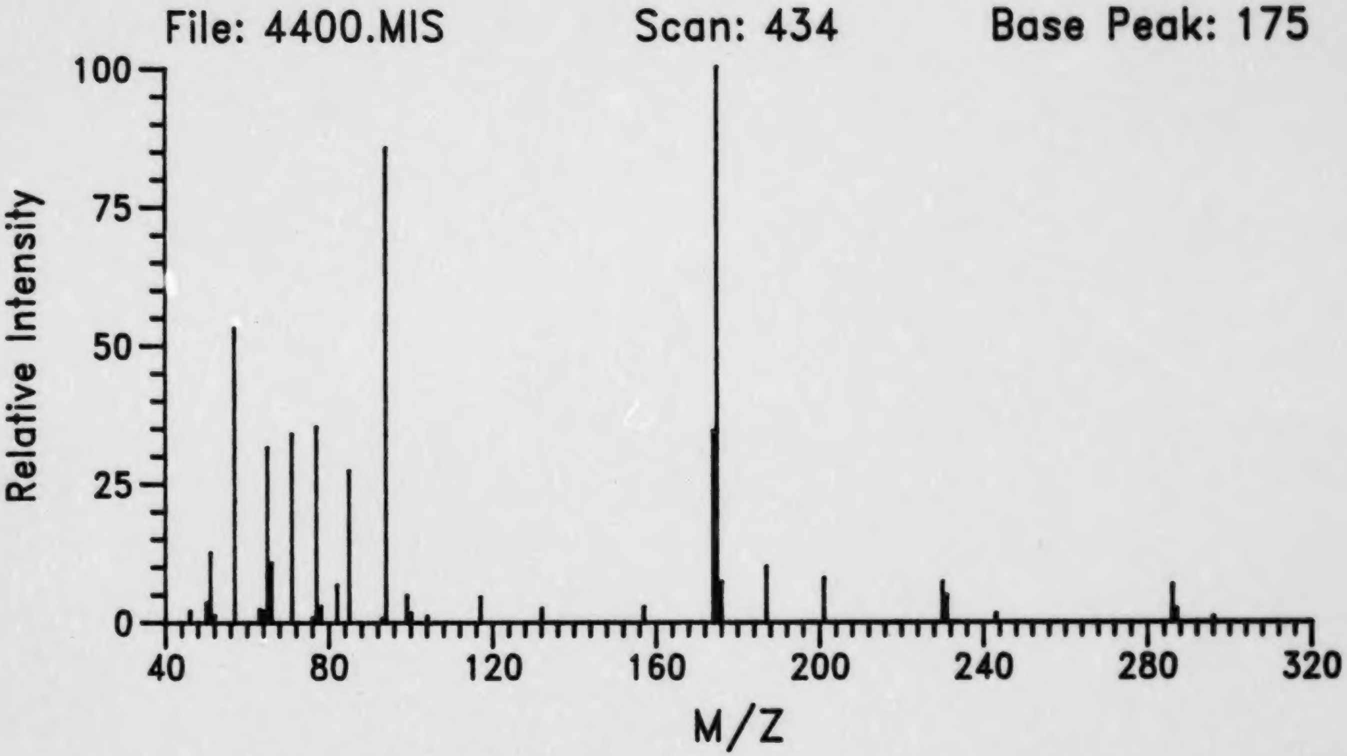


Figure B-63. Extract 4400 (base/neutral), auto and other laundries industry. Frequency of occurrence: 15.

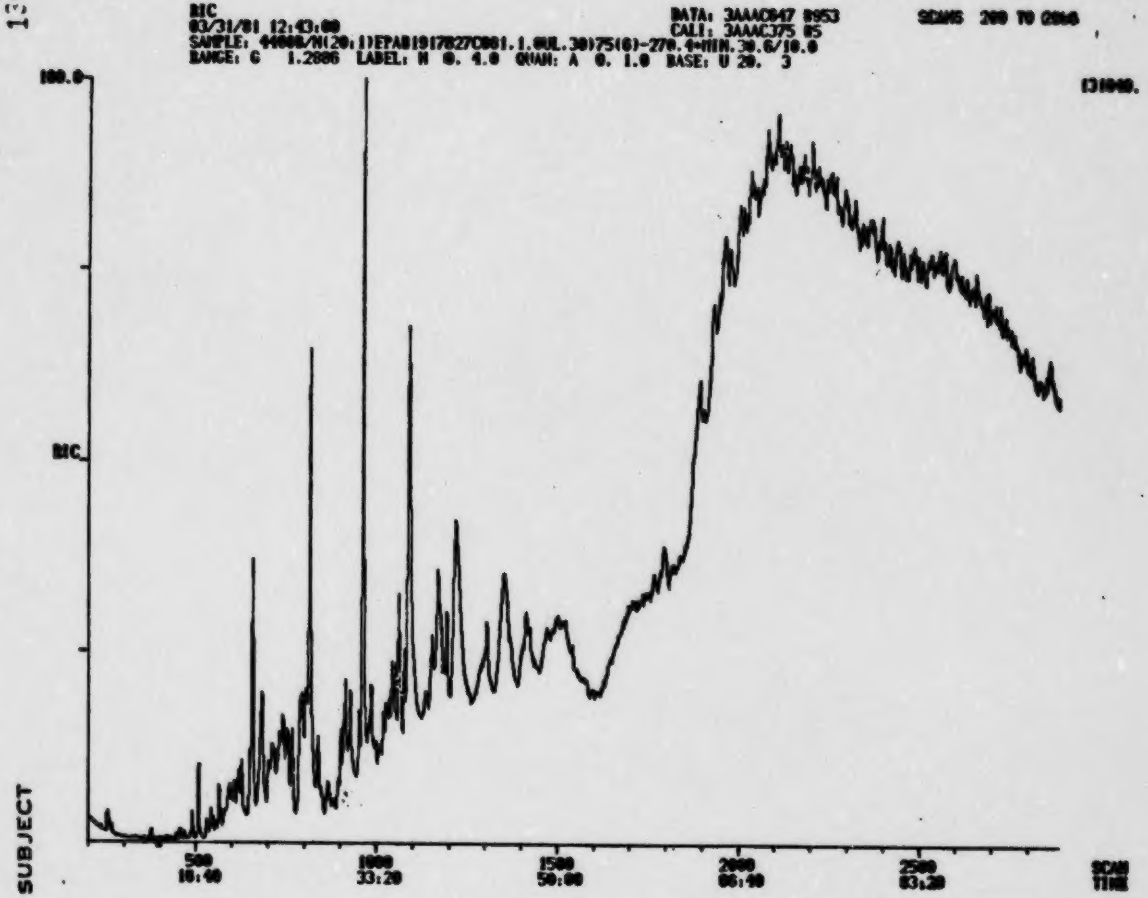


Figure B-64. Capillary chromatogram, extract 4400 (base/neutral), auto and other laundries industry.

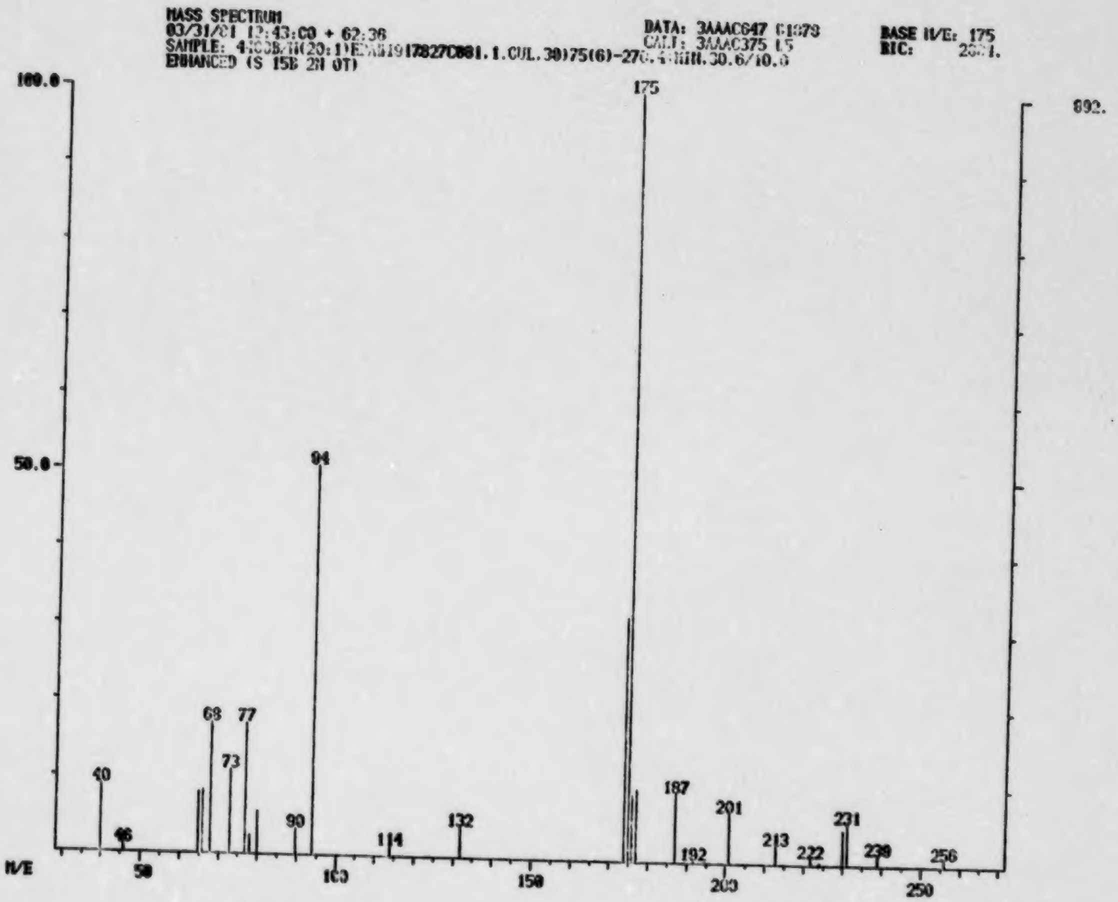


Figure B-65. EPA compound of interest, extract 4400 (base/neutral), auto and other laundries industry, one isomer, scan #1878.

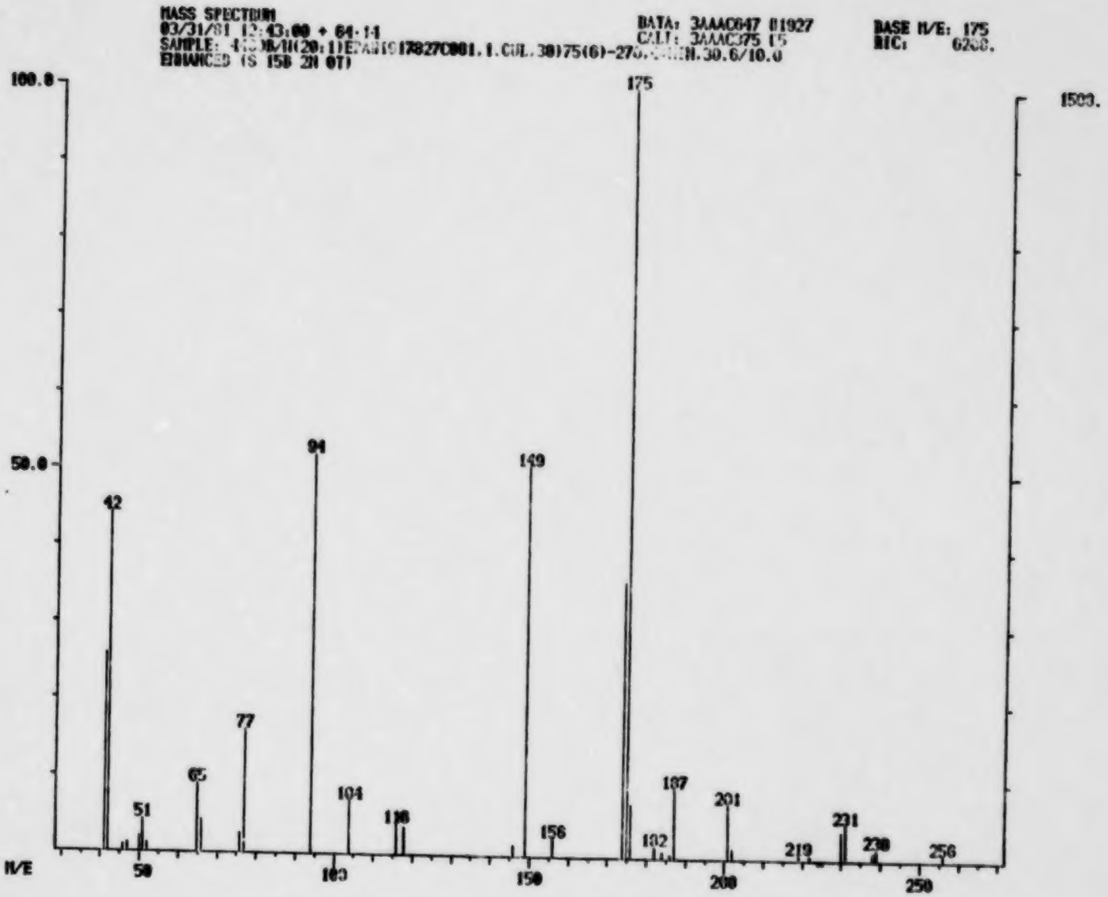


Figure B-66. EPA compound of interest, extract 4400 (base/neutral), auto and other laundries industry, one isomer, scan #1927.

Figure B-67. Conclusions

The mass spectrum of the EPA compound of interest is obtained, even to the last small ions barely above the baseline. However, application of chemical ionization techniques does not produce an ion of higher mass than the ions observed. If all of these ions belong to the same compound, then the ion of mass 256 cannot be a molecular ion, since there is no transition to mass 231 (25 mass units is not a possible loss). Accurate mass measurement on a successful chemical ionization determination would be the most desirable piece of information, but since chemical ionization was unsuccessful, the accurate mass measurement was not performed and no structural information is available.

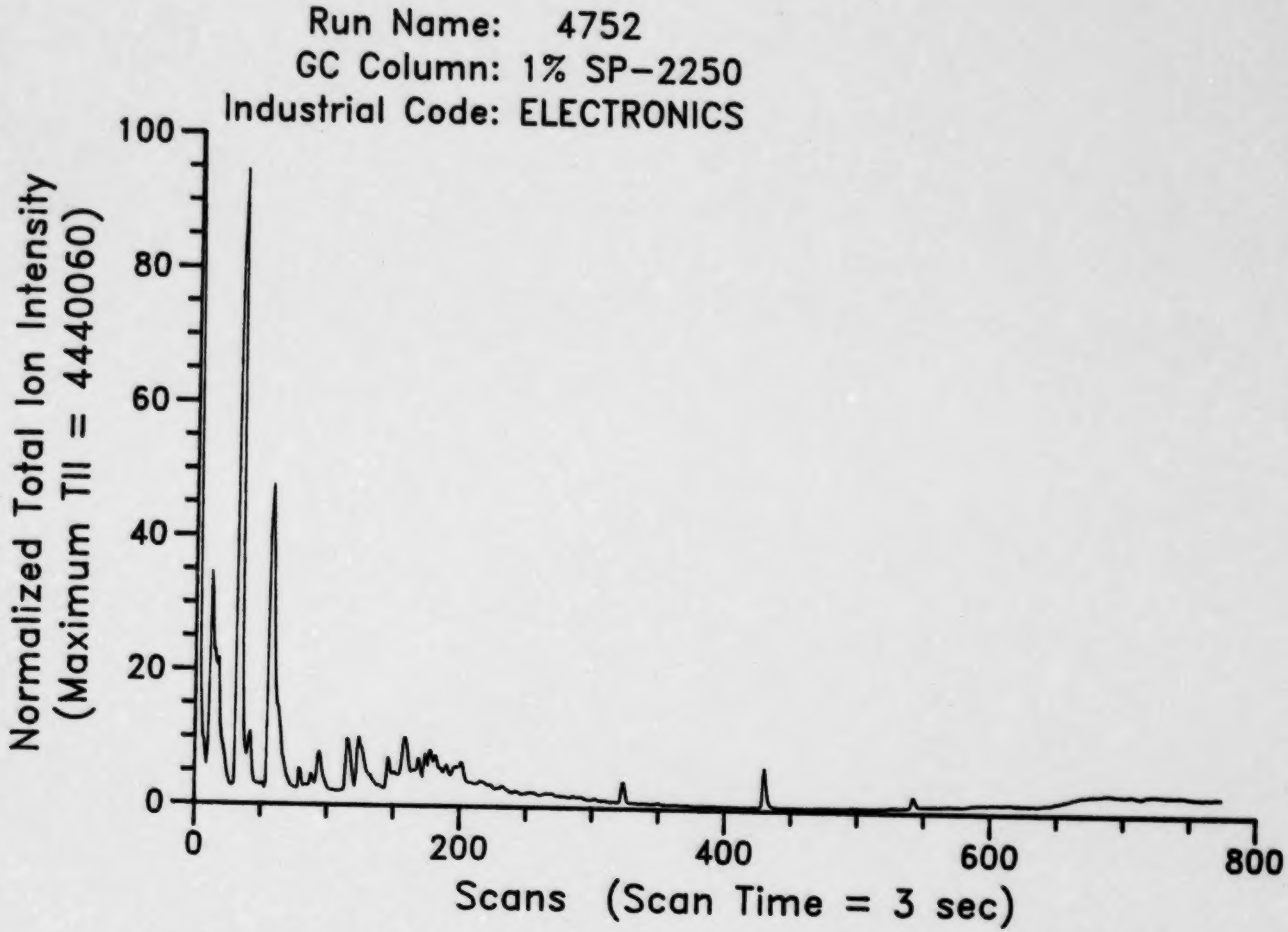


Figure B-68. Packed column chromatogram, extract 4752 (base/neutral), electronics industry.

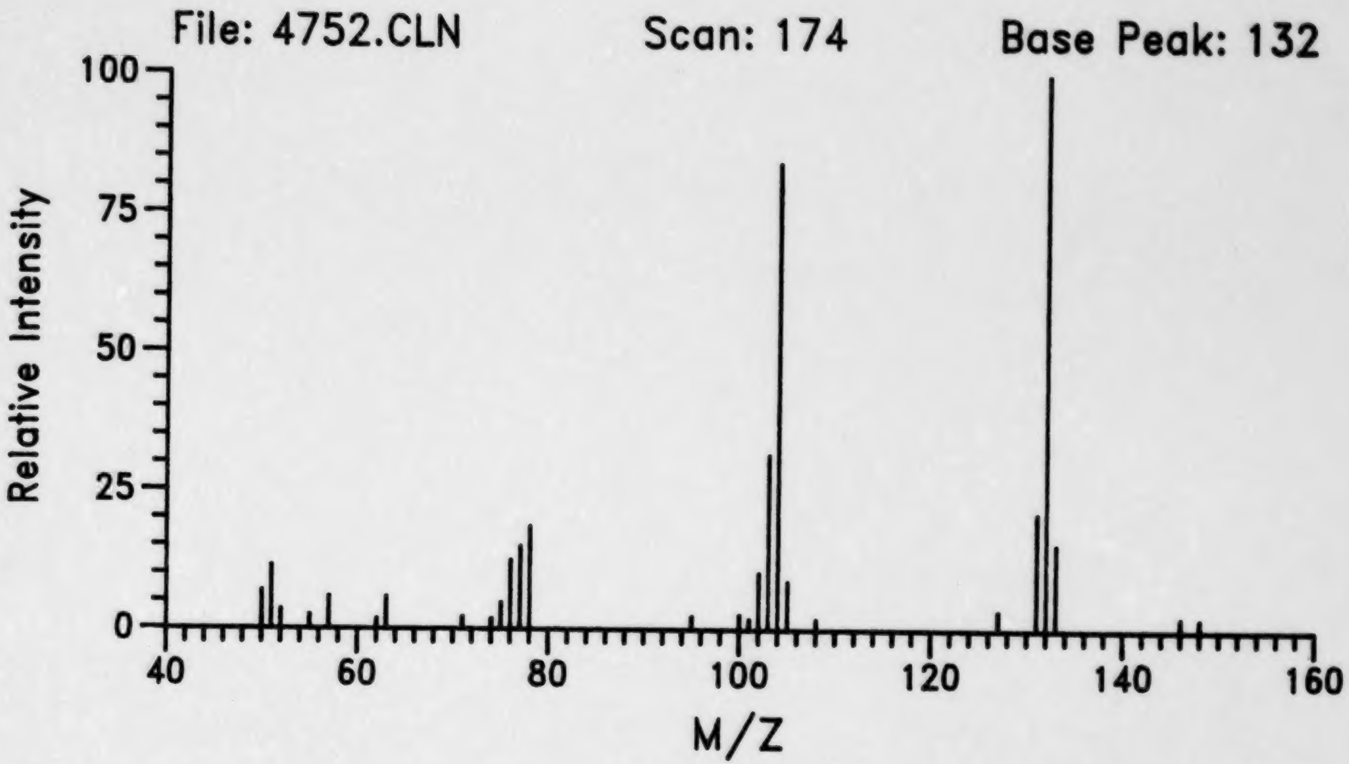


Figure B-69. Extract 4752 (base/neutral), electronics industry. Frequency of occurrence: 5.

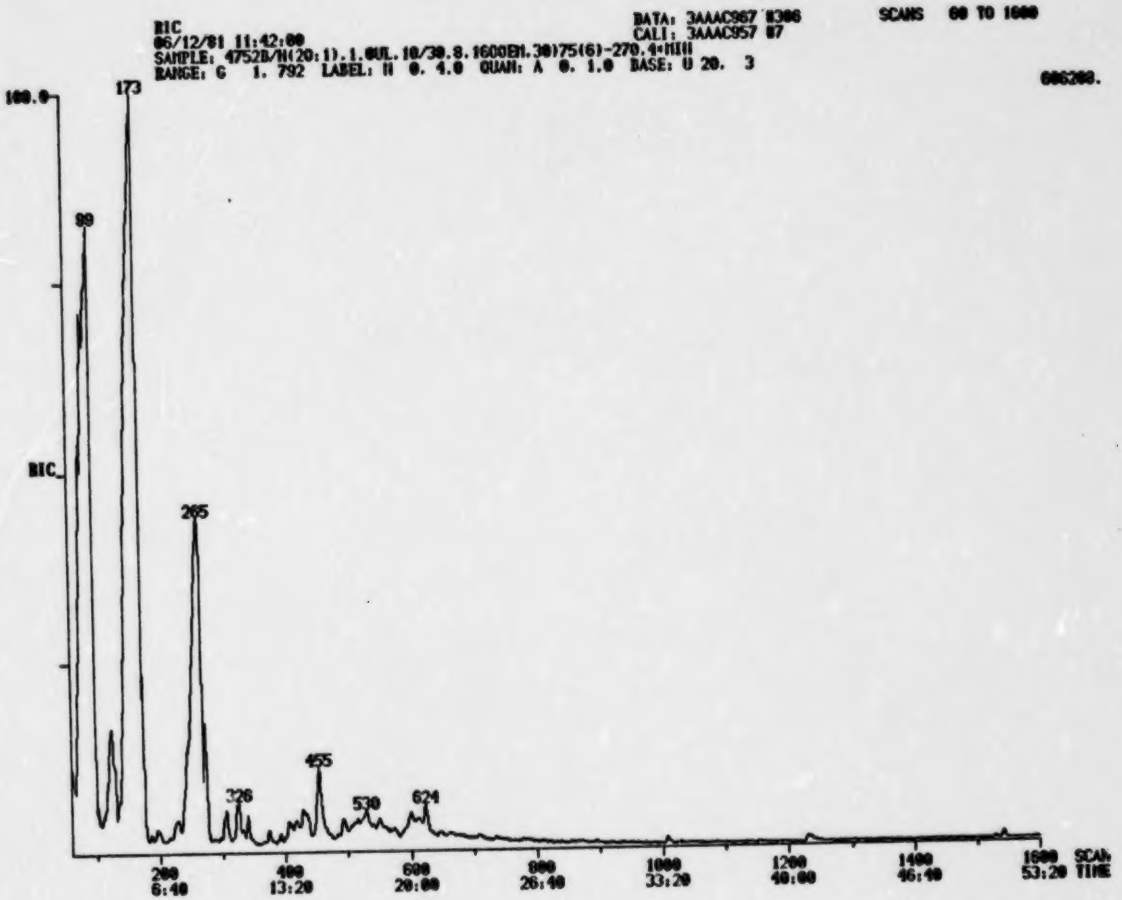


Figure B-70. Capillary chromatogram, extract 4752 (base/neutral), electronics industry.

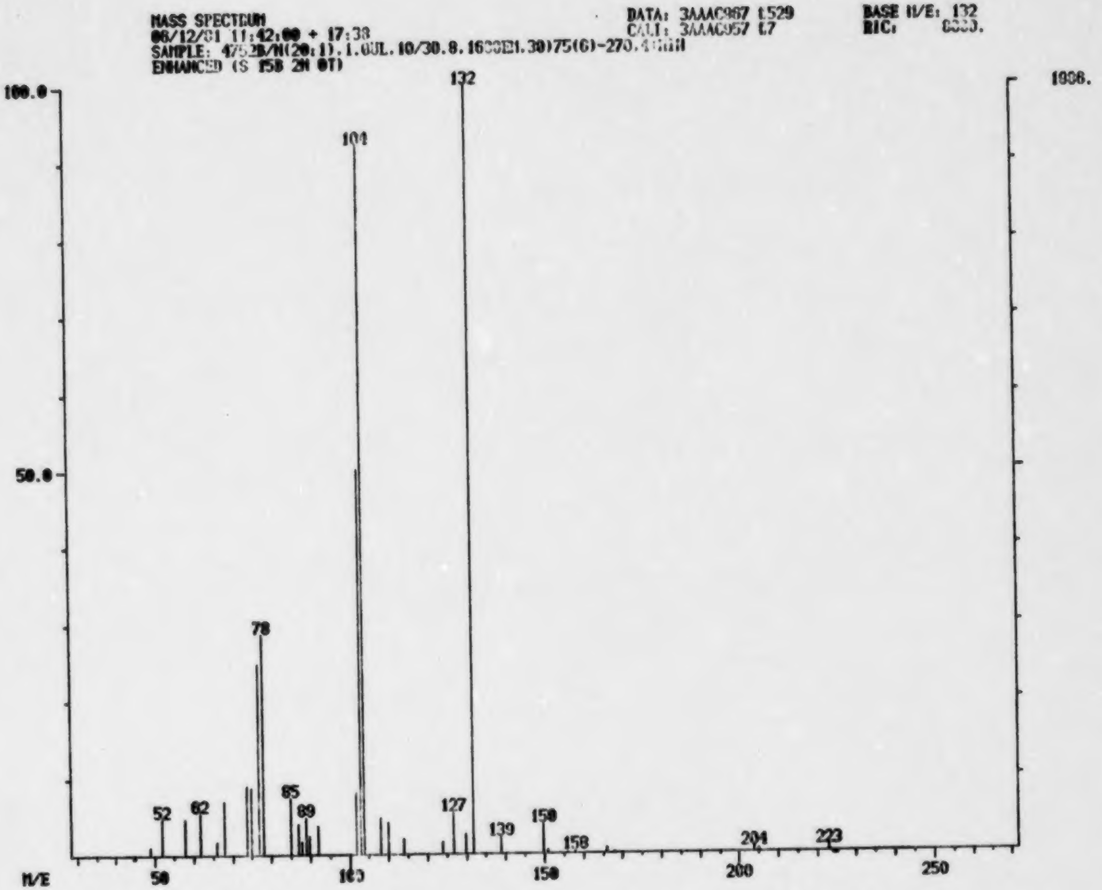
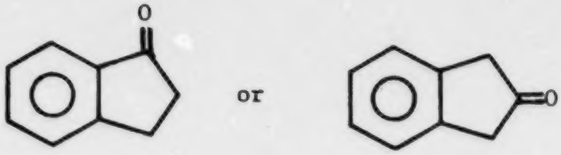


Figure B-71. EPA compound of interest, extract 4752 (base/neutral), electronics industry, scan #529.

Figure B-72. Conclusions

The appearance of the mass spectrum (with computerized library search) and accurate mass measurement and chemical ionization data are consistent with structural assignment as an indanone isomer.



Accordingly, the 1-and 2-indanones were purchased and analyzed. The elution of the standards is approximately 500 scan (~7-8 min) earlier than the election of the compound of interest. The compound of interest either: (1) is not an indanone isomer; or (2) is being formed, perhaps on-column, from other component(s) of the extract.

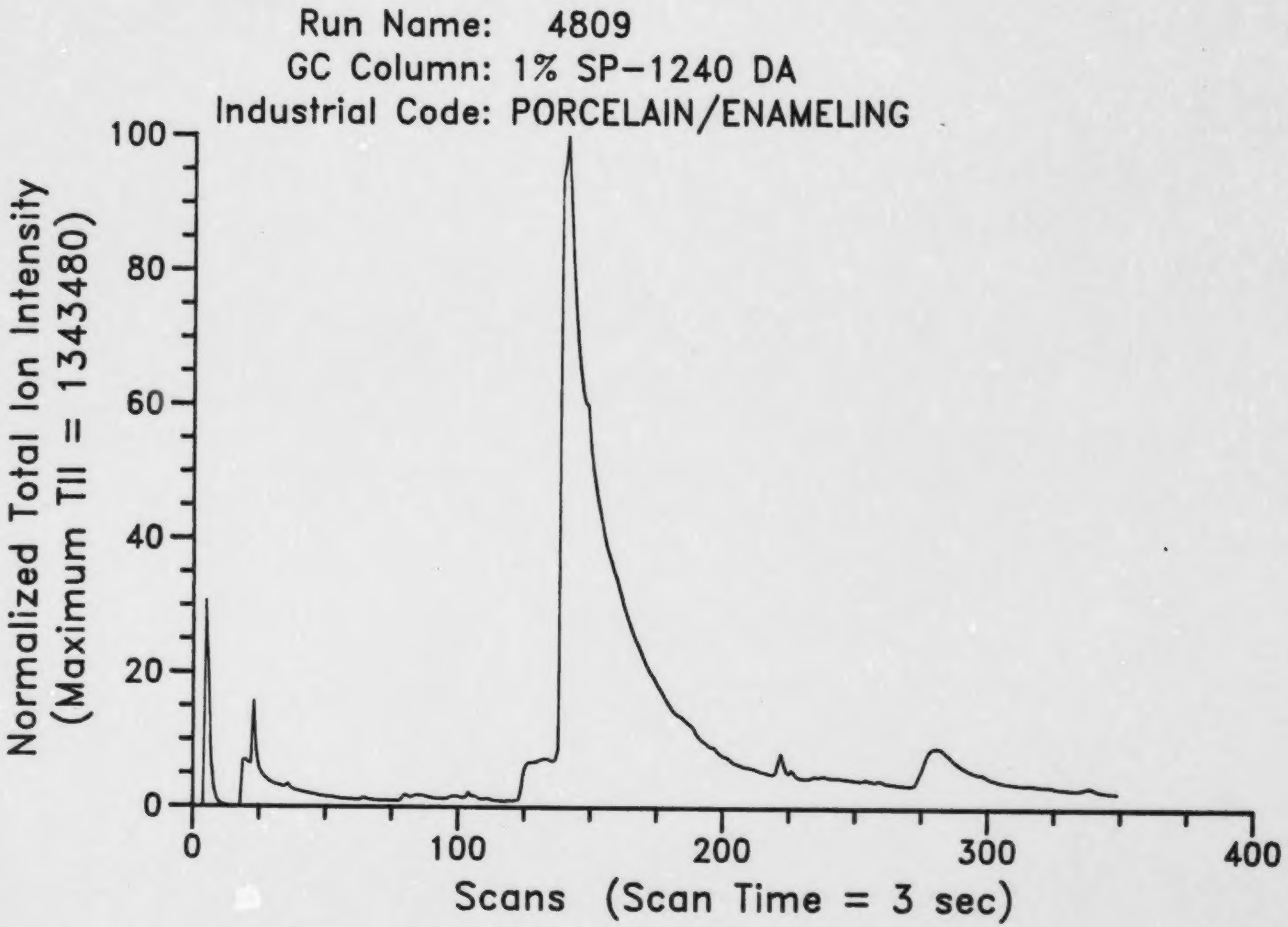


Figure B-73. Packed column chromatogram, extract 4809 (acid), porcelain/enameling industry.

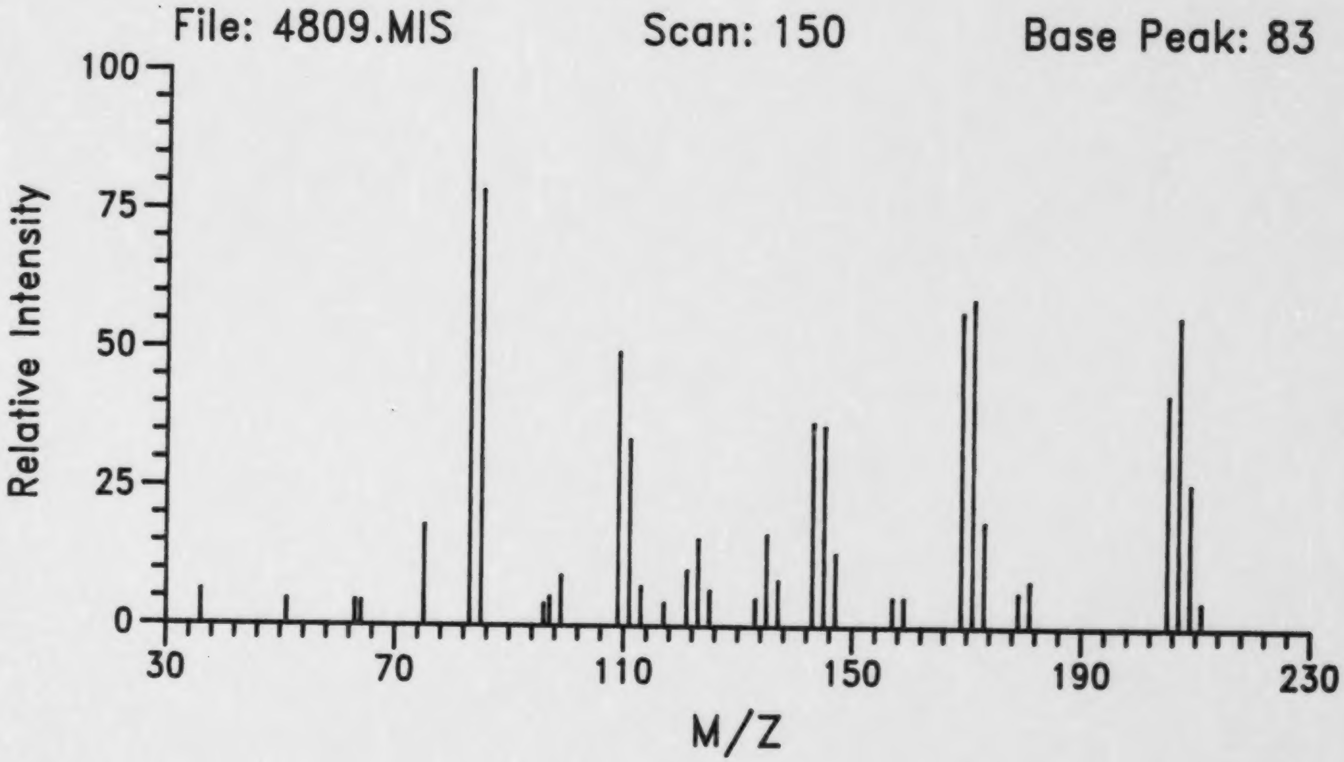


Figure B-74. Extract 4809 (acid), porcelain/enameling industry.
Frequency of occurrence: 15.

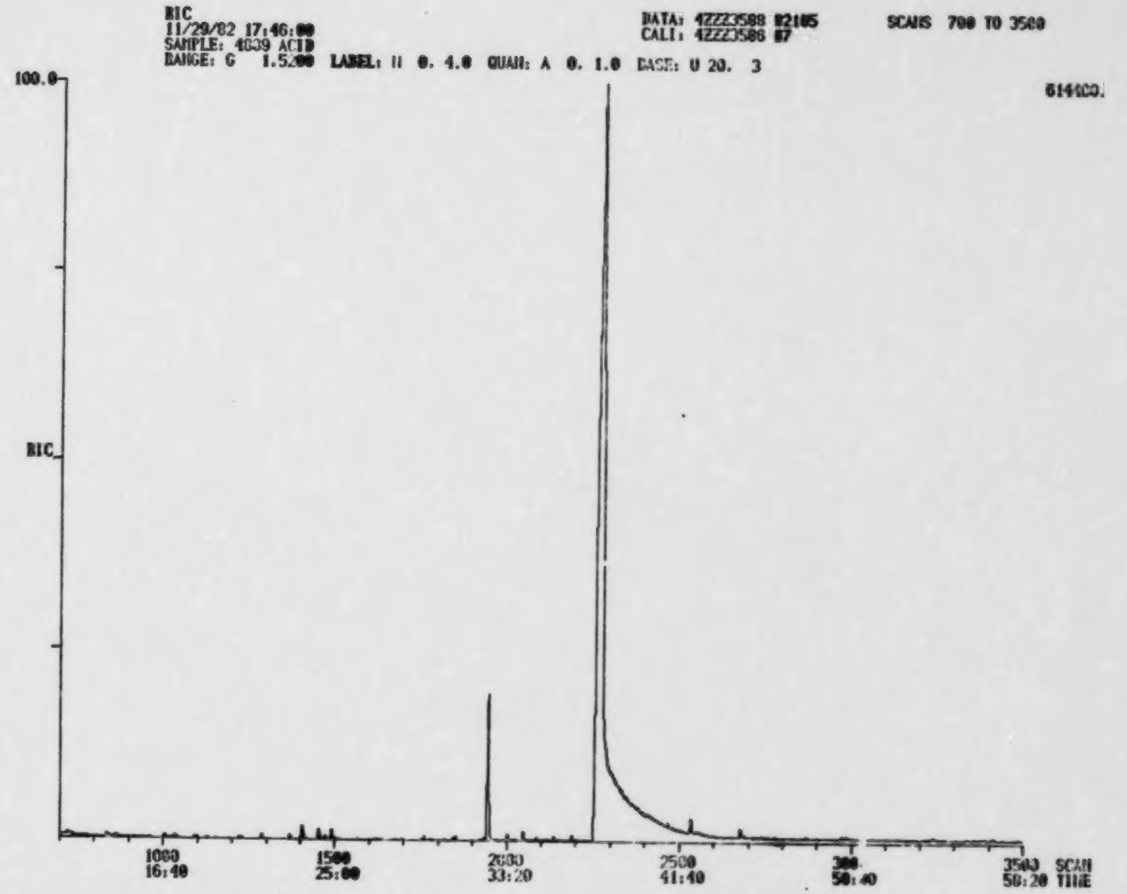


Figure B-75. Capillary chromatogram, extract 4809(acid), porcelain/enameling industry.

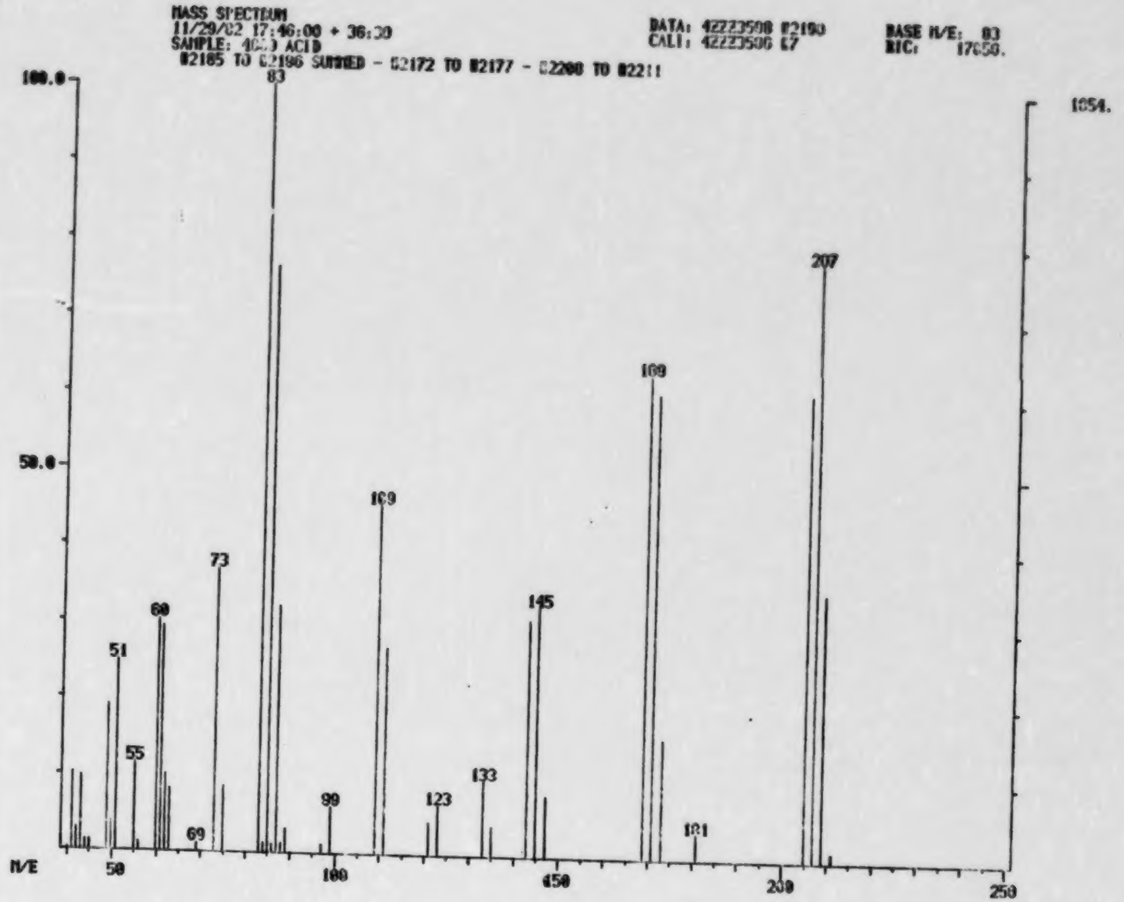


Figure B-76. EPA compound of interest, extract 4809 (acid), porcelain/enameling industry, scan #2190.

Figure B-77. Conclusions

The chlorine isotope cluster at mass 205/207/209/211 is characteristic of four chlorine atoms. The accurate mass measurement data support this assignment, with a composition for this ion of $C_5H_5Cl_4$ (error, 4.2 mmu). Chemical ionization techniques do not disclose any ions of higher mass than this cluster which cannot, however, represent a molecular species. A molecular species would be C_5X_{10}, indicating either a ring or one site of unsaturation. The most probable molecular composition would thus be $C_5H_5Cl_5$, with no more definite structural assignment possible without standards.

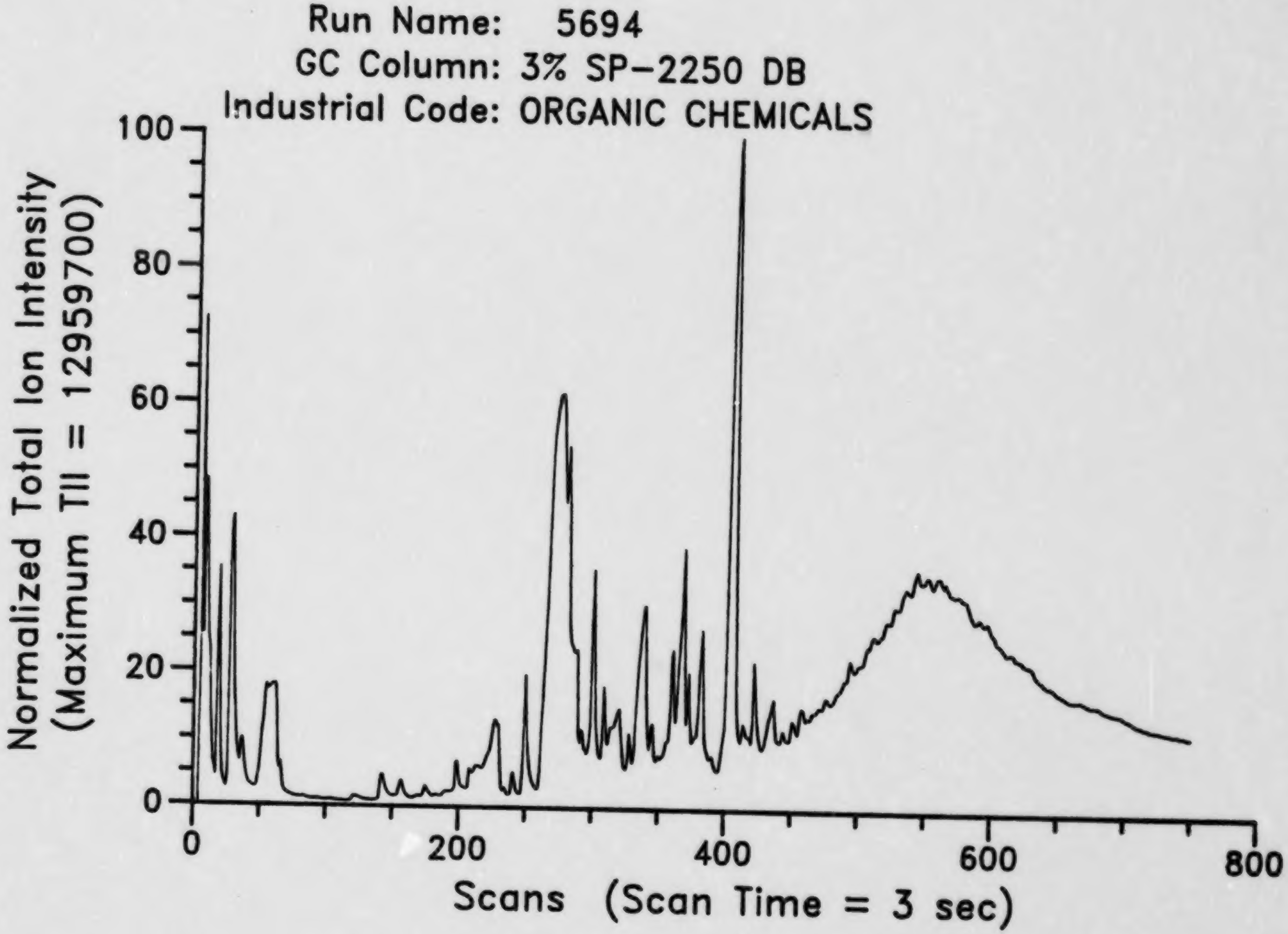


Figure B-78. Packed column chromatogram, extract 5694 (base/neutral), organic chemicals industry.

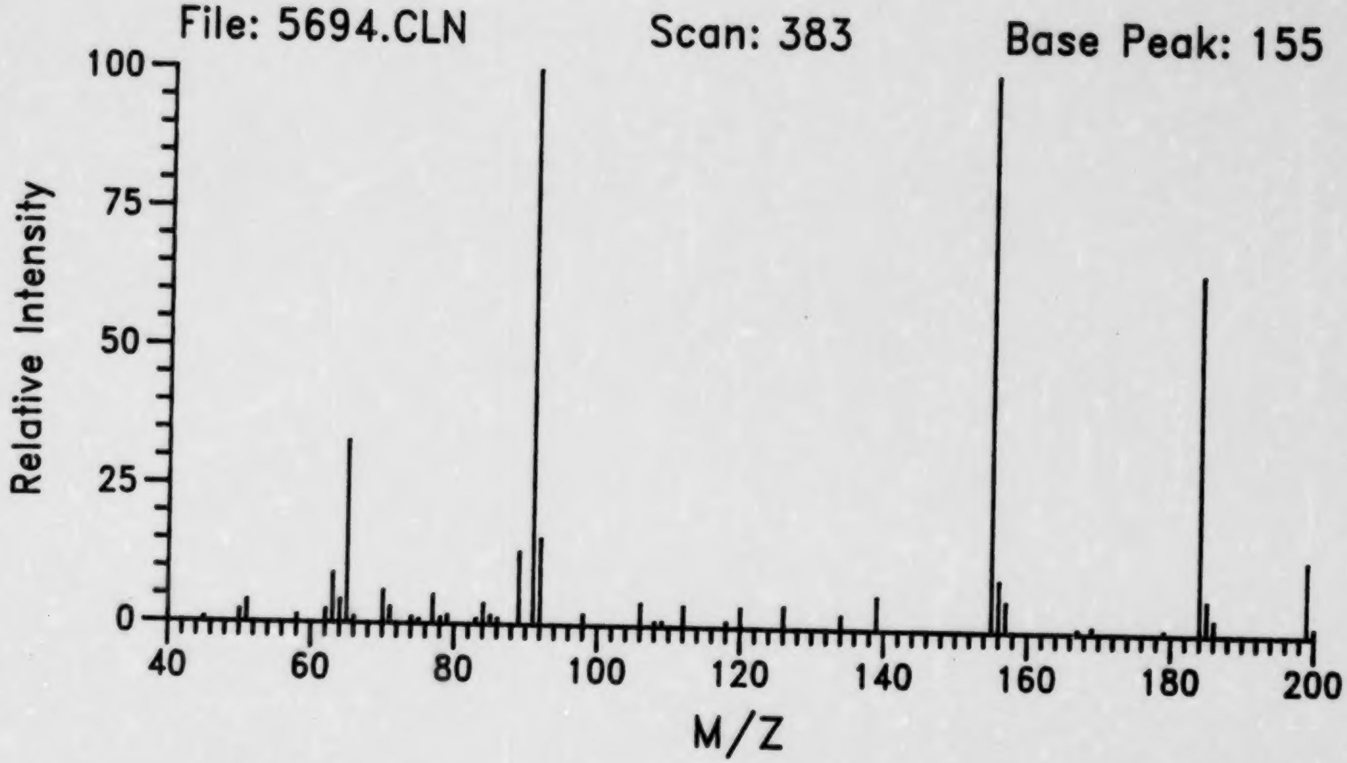


Figure B-79. Extract 5694 (base/neutral), organic chemicals industry. Frequency of occurrence: 17.

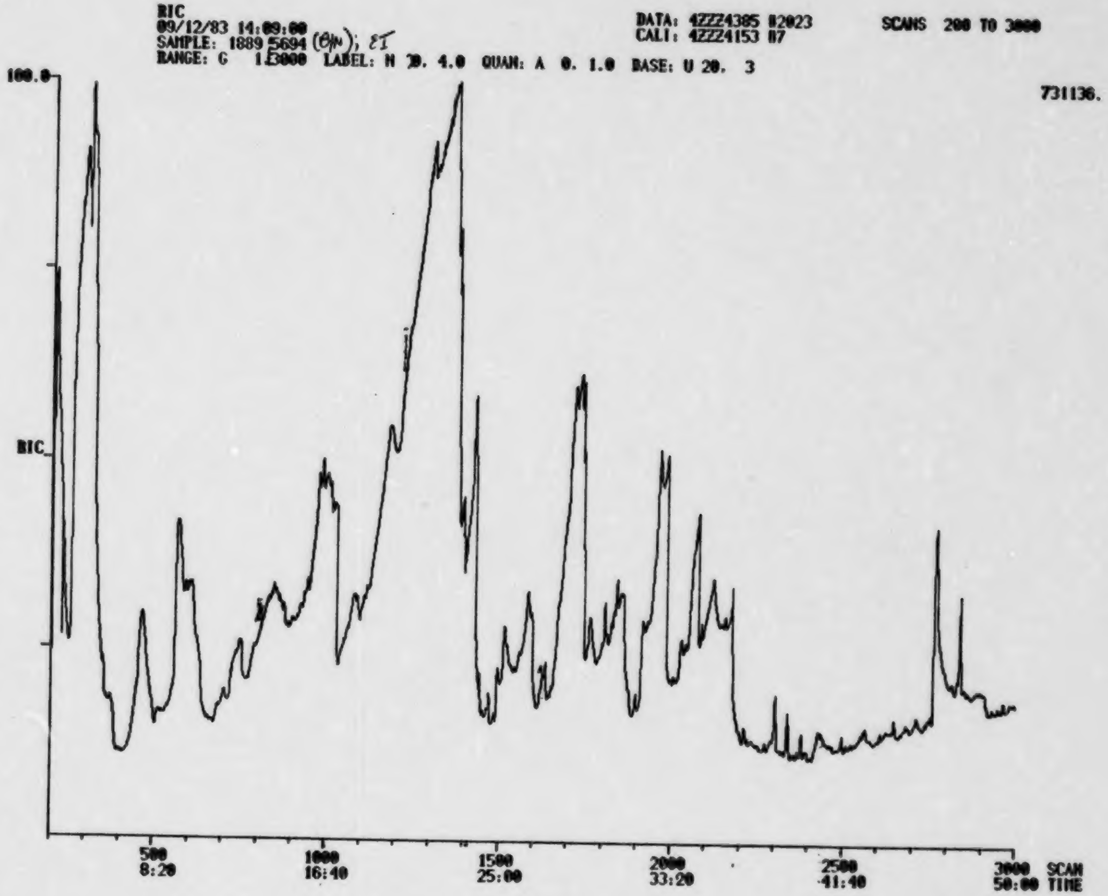


Figure B-80. Capillary chromatogram, extract 5694 (base/neutral), organic chemicals industry.

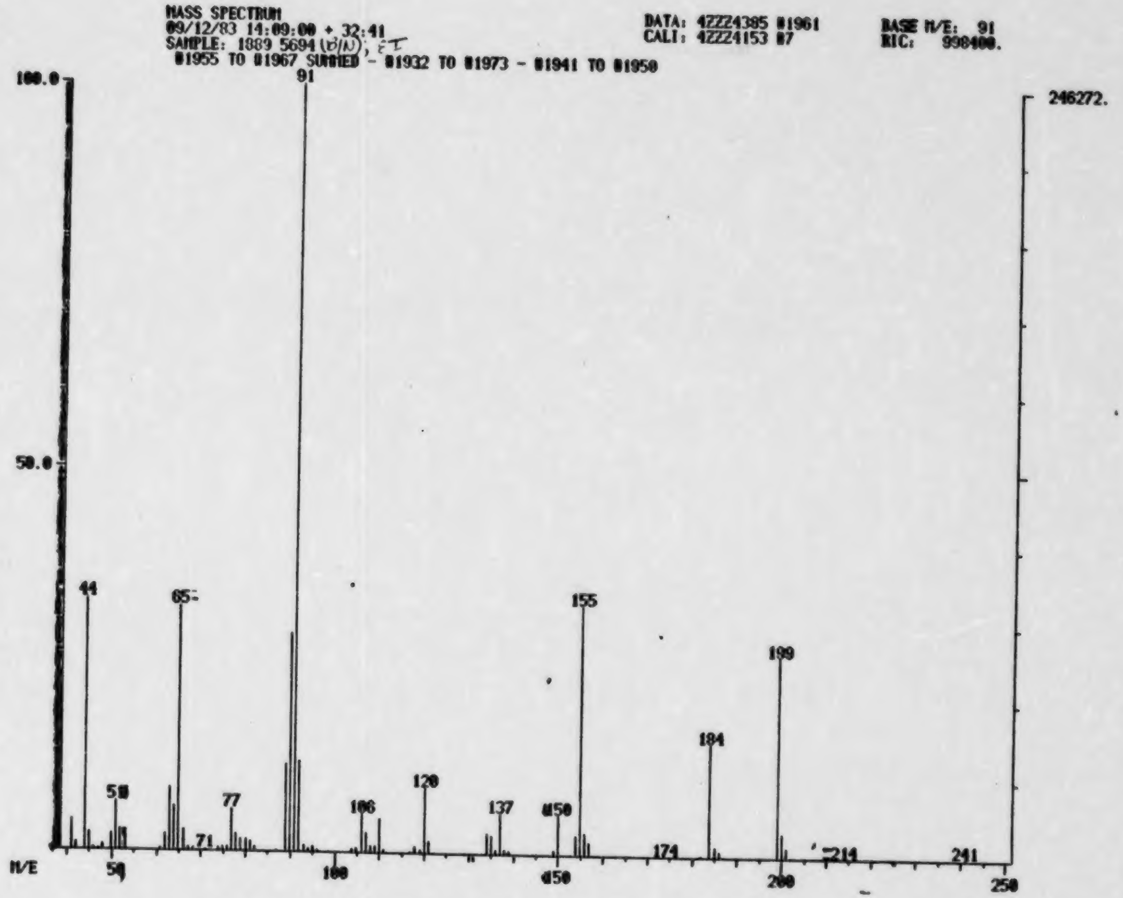


Figure B-81. Mass spectrum of EPA compound of interest, extract 5694 (base/neutral), organic chemicals industry, one isomer, scan #1961.

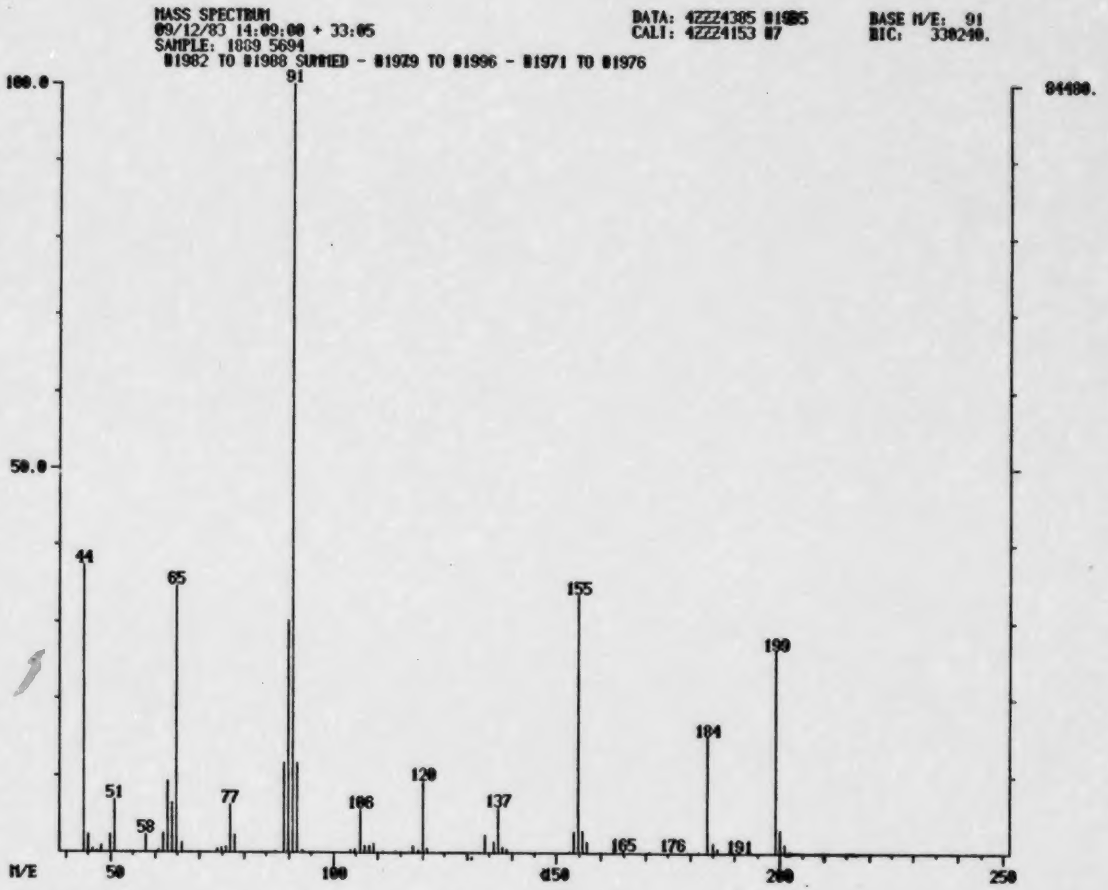


Figure B-82. Mass spectrum of EPA compound of interest, extract 5694 (base/neutral), organic chemicals industry, one isomer, scan #1985.

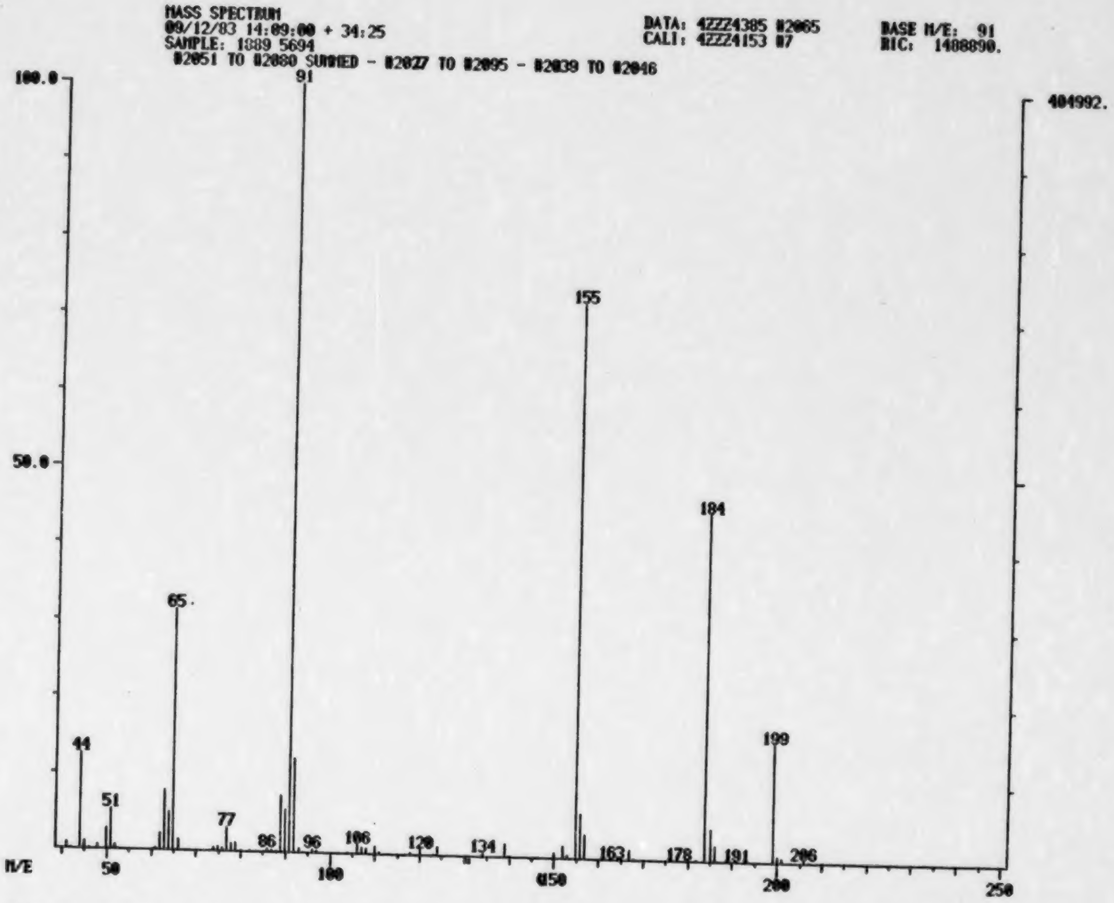


Figure B-83. Mass spectrum of EPA compound of interest, extract 5694 (base/neutral), organic chemicals industry, one isomer, scan #2065.

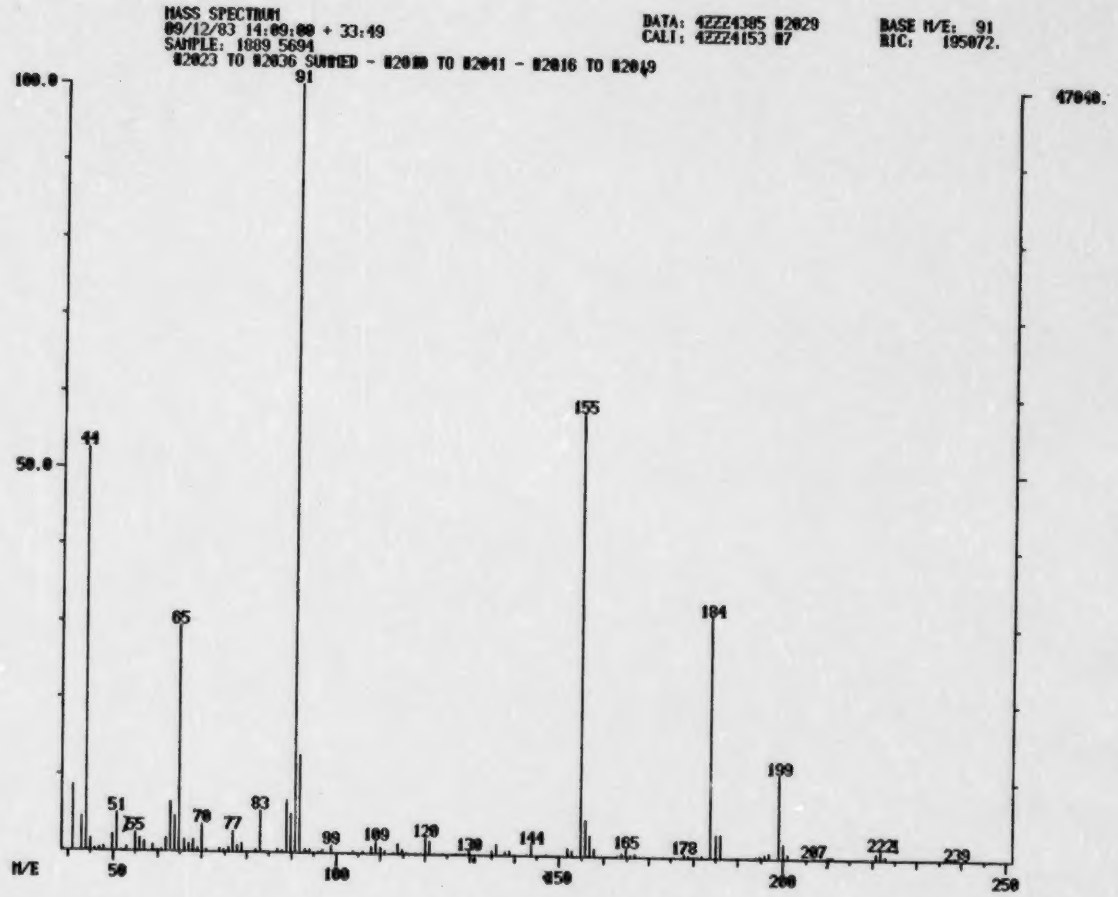


Figure B-84. Mass spectrum of EPA compound of interest, extract 5694 (base/neutral), organic chemicals industry, one isomer, scan #2029.

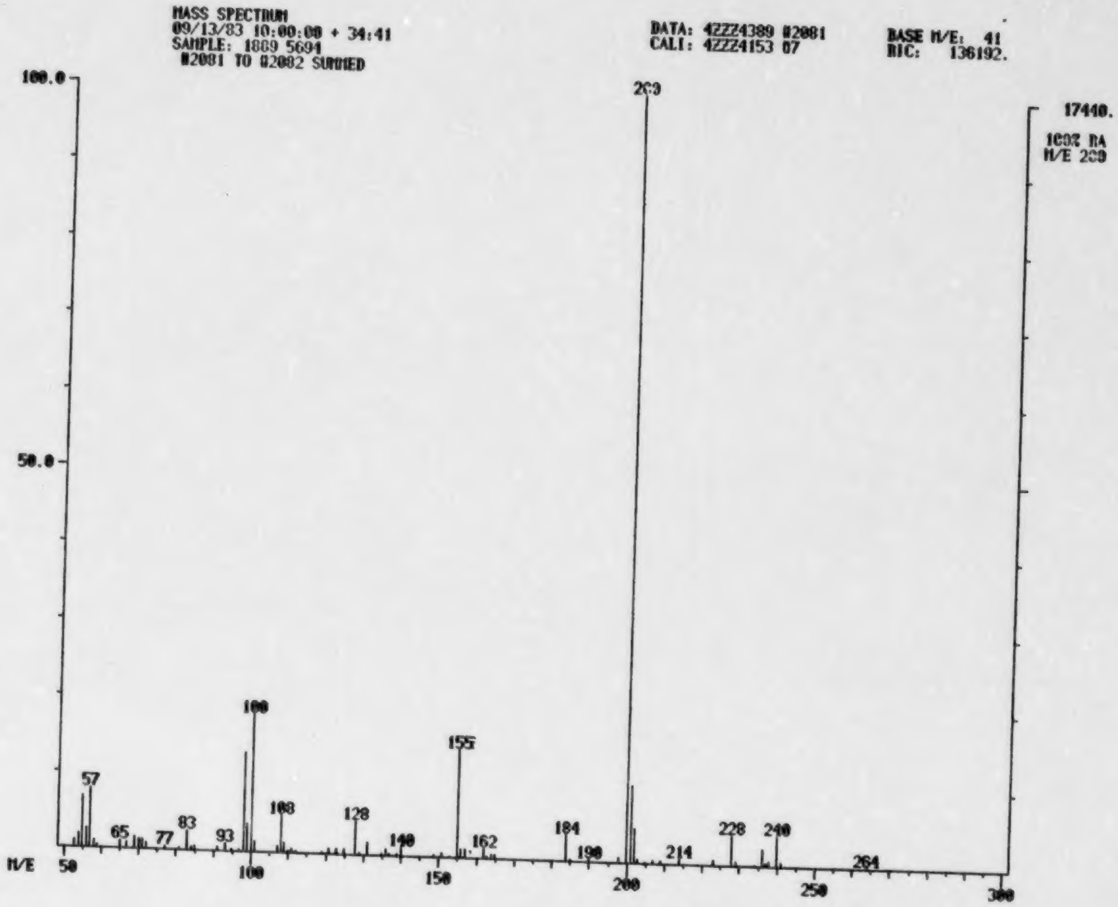
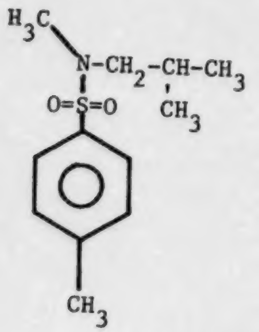


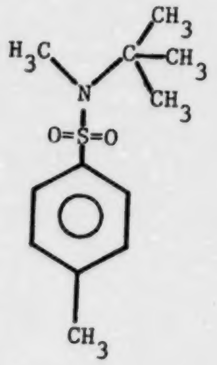
Figure B-85. Representative isobutane chemical ionization mass spectrum of EPA compound of interest, scan #2081.

Figure B-86. Conclusions

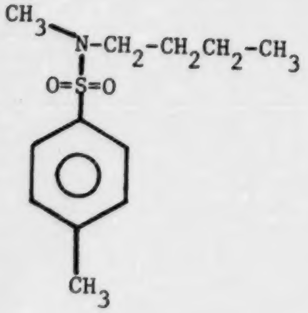
Computerized library search identifies the compound of interest as a sulfonamide (three of the first five choices), but the FIT is poor, indicating that the exact compound is not present in the library. Accurate mass measurement verifies the assignment as a sulfonamide (m/z 199, $C_{19}H_{13}SO_2N$; error, -6.2 mmu). Chemical ionization (isobutane) mass spectrometry shows the highest mass peak at m/z 240, most probably an $(M-1)^+$ species. Tentative structure assignments are presented below:

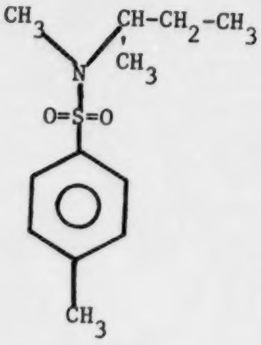

H3C
N-CH2-CH-CH3
O=S=O
CH3
CH3

or


CH3
H3C
C-CH3
N
CH3
O=S=O
CH3

or


CH3
N-CH2-CH2CH2-CH3
O=S=O
CH3


CH3
CH-CH2-CH3
CH3
N
O=S=O
CH3

Note the presence of
m/z 214 in one EI spectrum

Since four compounds are present at four distinct retention times, all of these possibilities are viable. However, no standards are available. Capillary GC/FTIR would be valuable in assigning structures, but not enough compound is available.

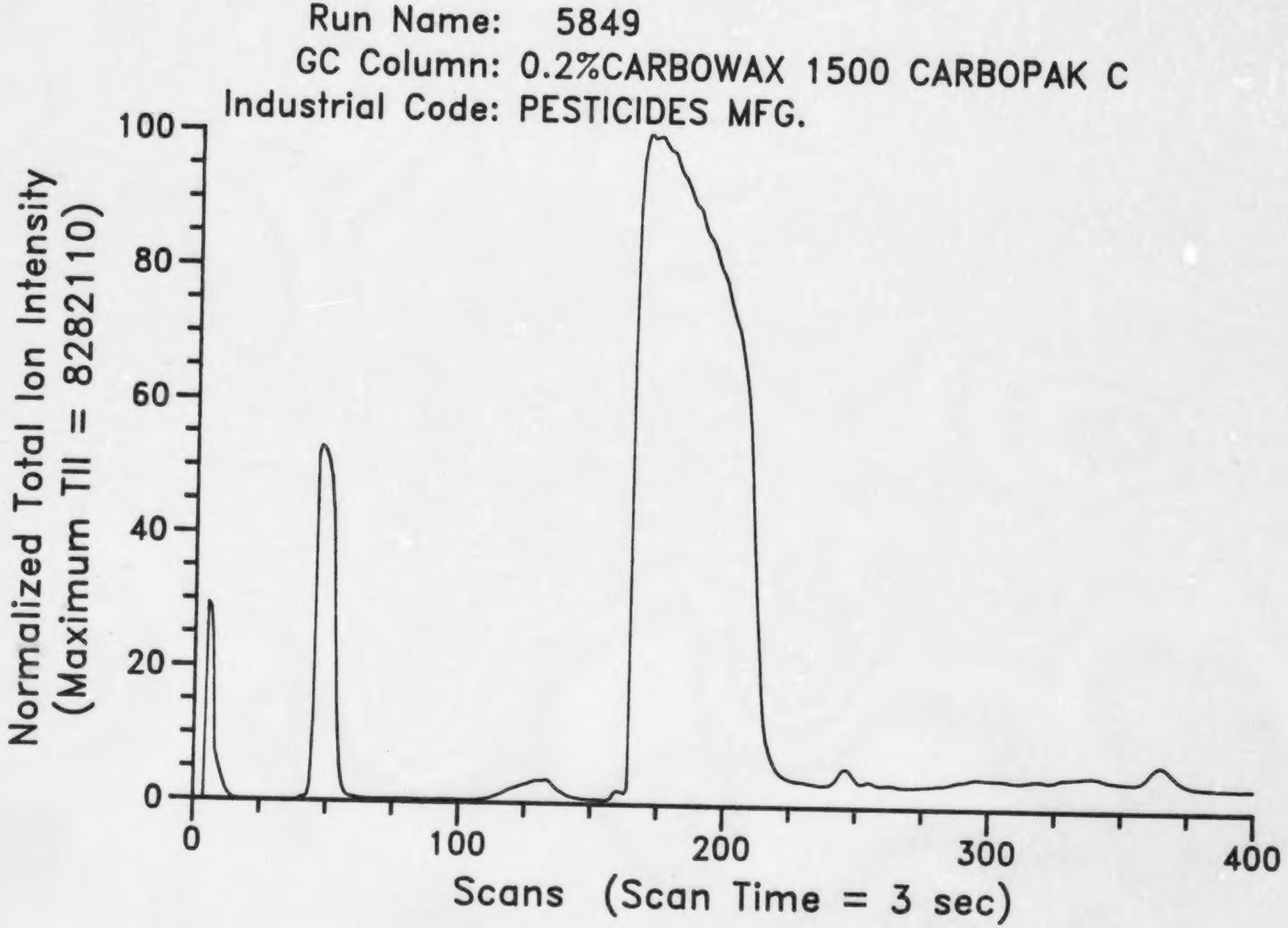


Figure B-87. Packed column chromatogram, extract 5894 (acid), pesticides manufacturing industry.

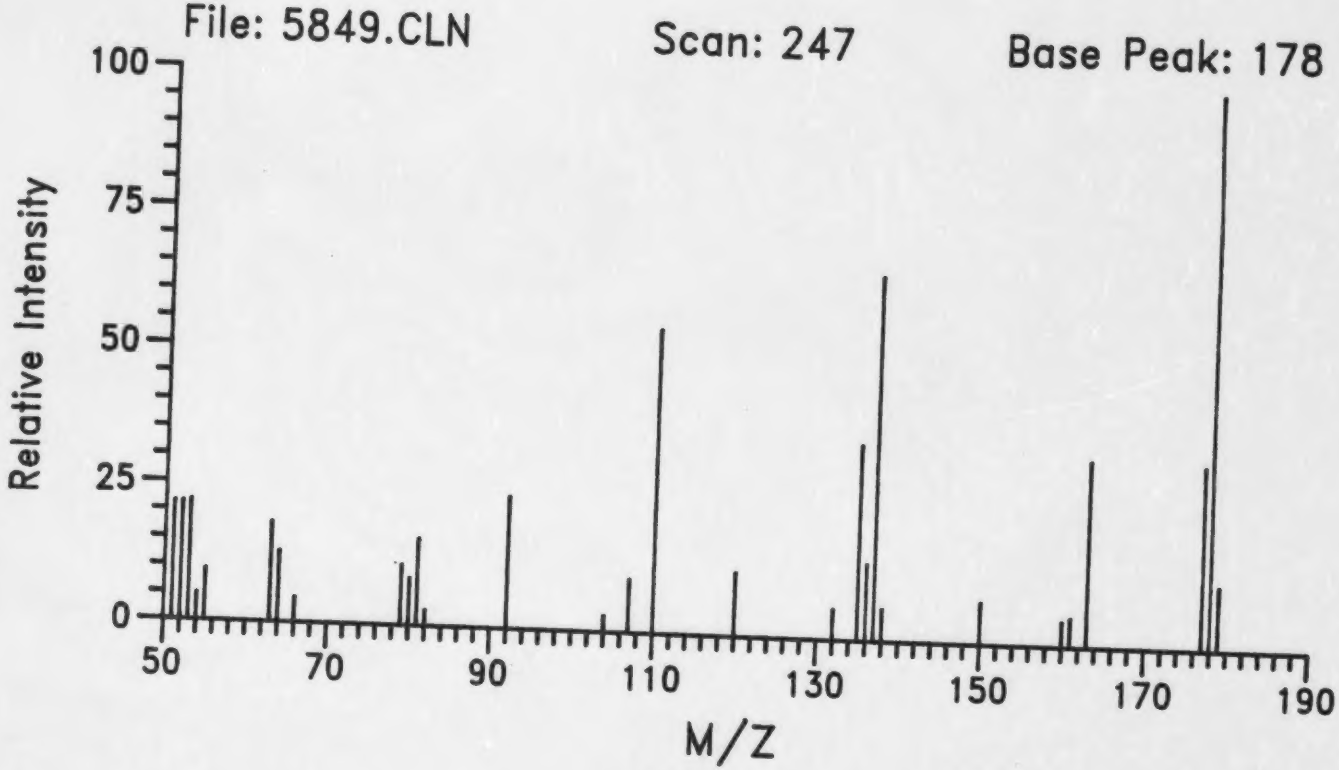


Figure B-88. Extract 5849 (acid), pesticides manufacturing industry. Frequency of occurrence: 5.

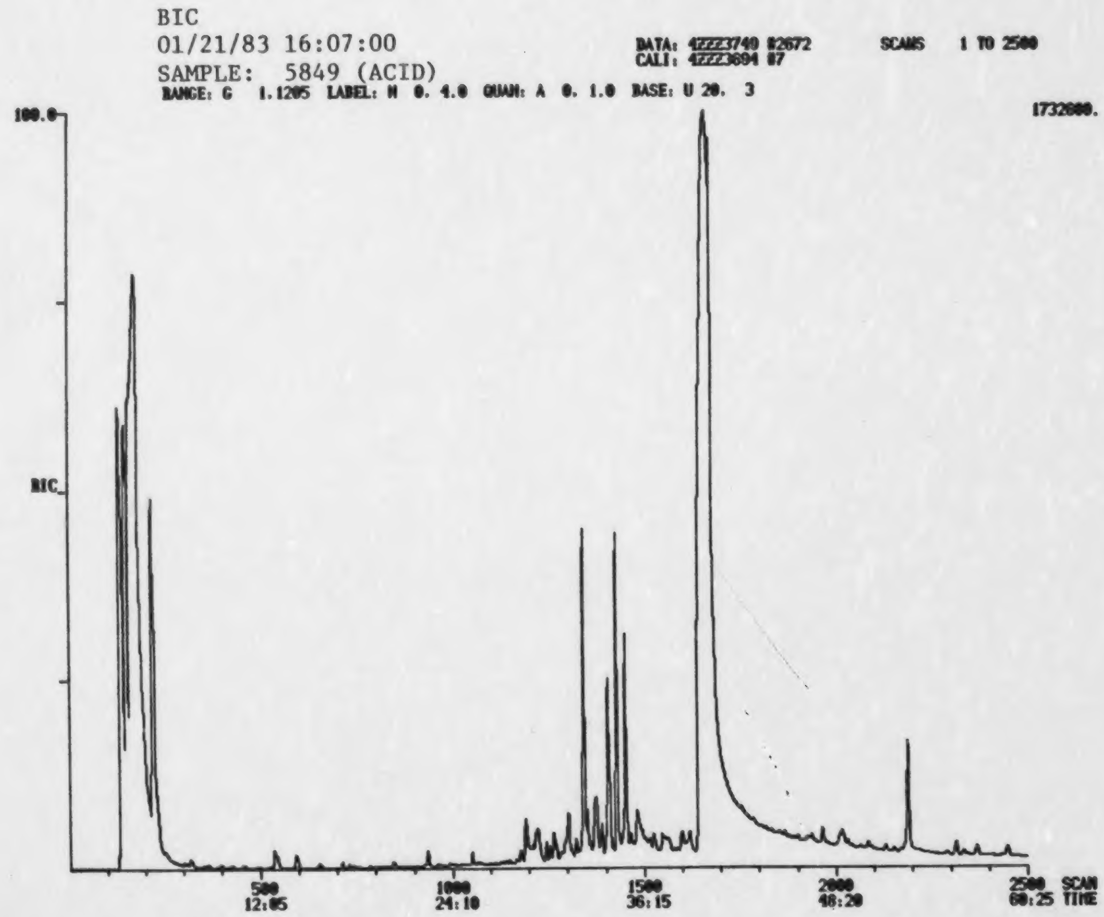


Figure B-89. Capillary chromatogram, extract 5849 (acid), pesticides manufacturing industry.

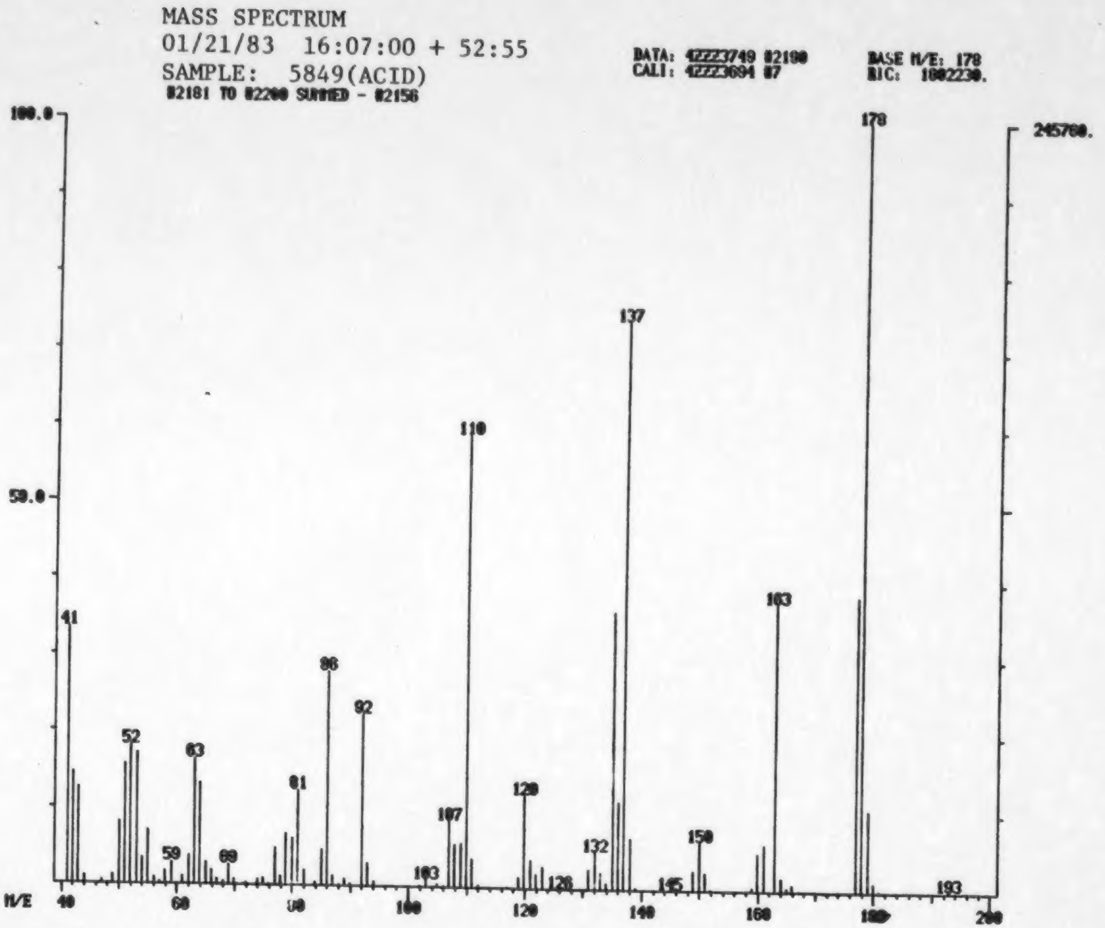


Figure B-90. EPA compound of interest, extract 5894 (acid), pesticides manufacturing industry, scan #2190.

Figure B-91. Conclusions

Accurate mass measurement data support an assignment of $C_5H_{10}N_2O_5$ (error, 0.8 mmu) for the composition of the molecular ion. Only a few structural features can be deduced from the mass spectrum: $(M-15)^+$ implies at least one methyl group; $(M-41)^+$ results from loss of C_3H_5; $(M-43)^+$ results from loss of an acetyl group. The oxygen atoms do not appear to be present as hydroxy groups, since no loss of H_2O is observed. Functional group analysis (i.e., FT-IR) would be very useful but not enough material is present.

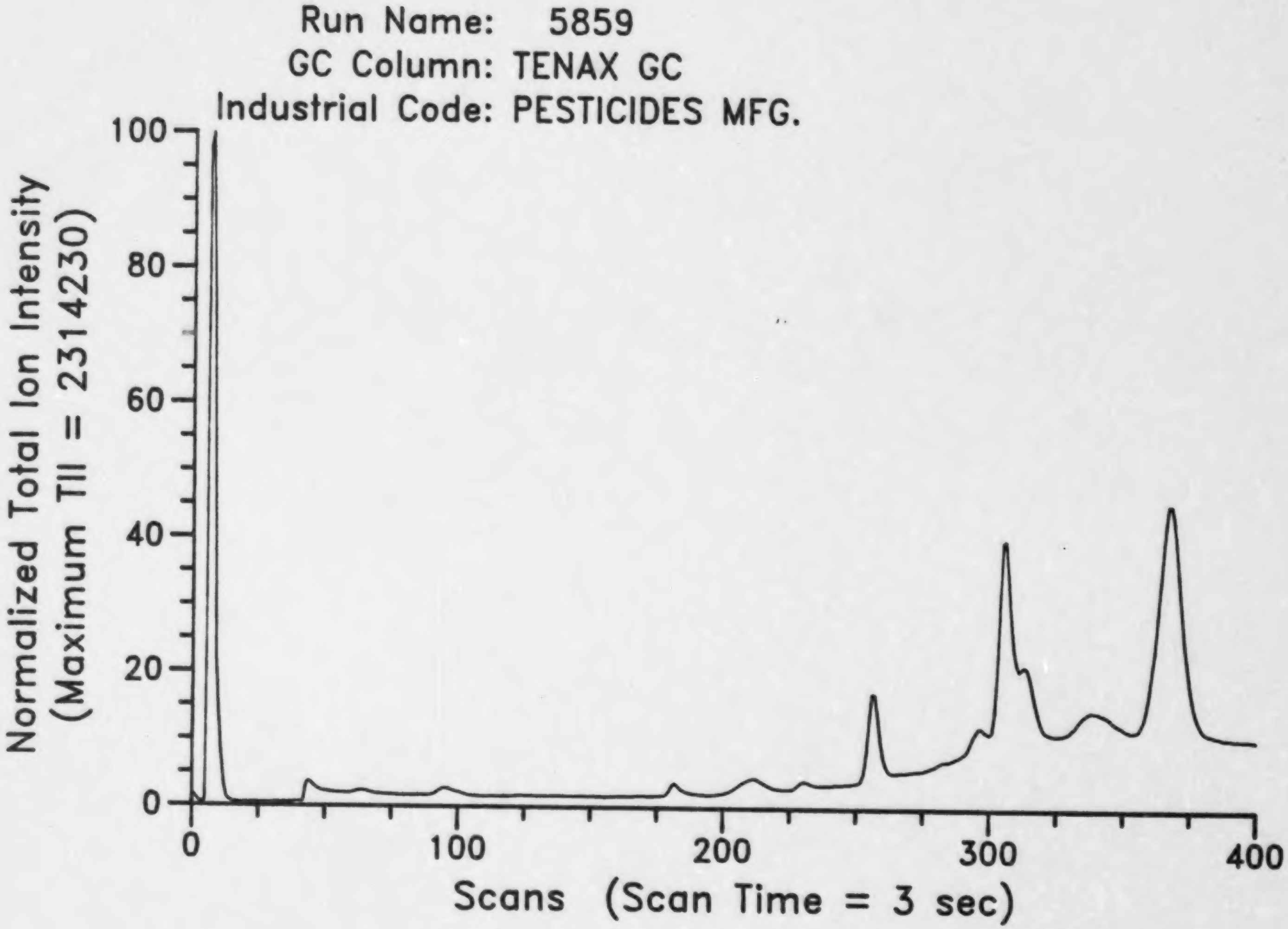


Figure B-92. Packed column chromatogram, extract 5859 (acid), pesticides manufacturing industry.

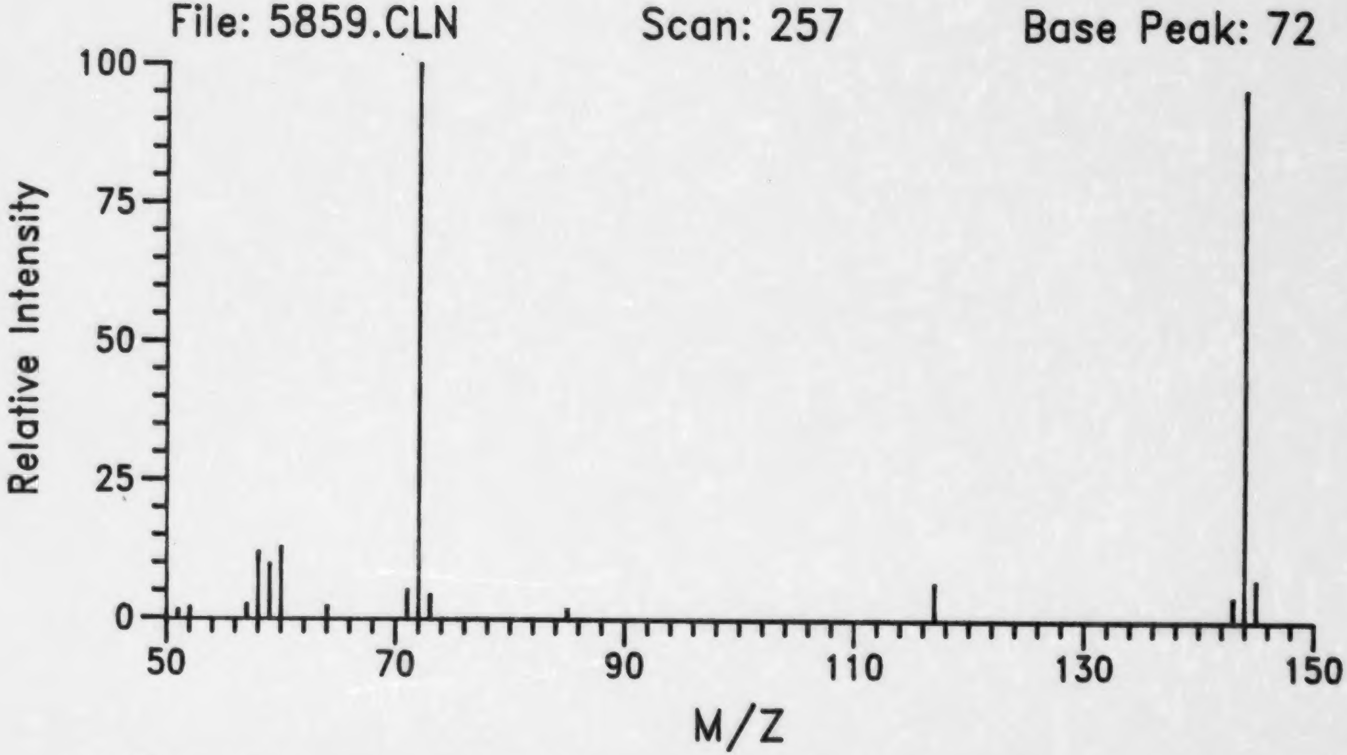


Figure B-93. Extract 5859 (acid), pesticides manufacturing industry. Frequency of occurrence: 7.

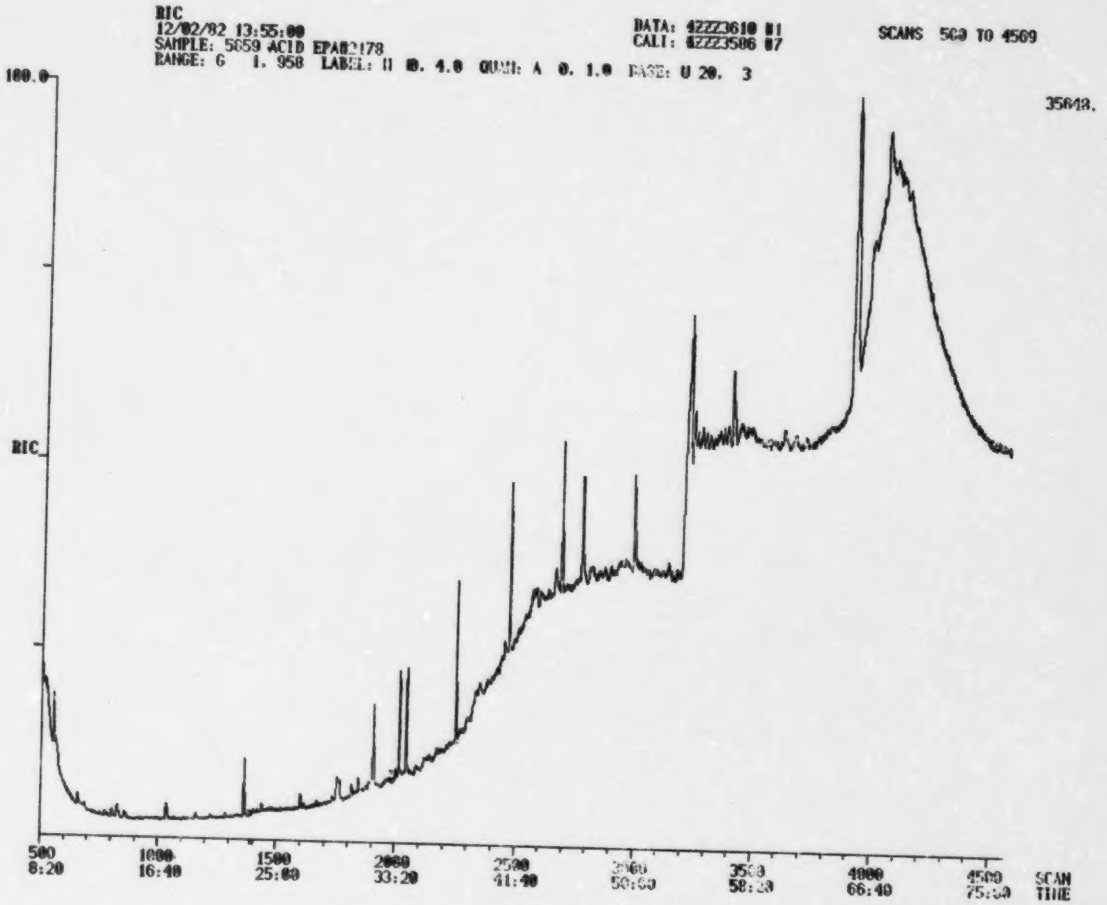


Figure B-94. Capillary chromatogram, extract 5859 (acid), pesticides manufacturing industry.

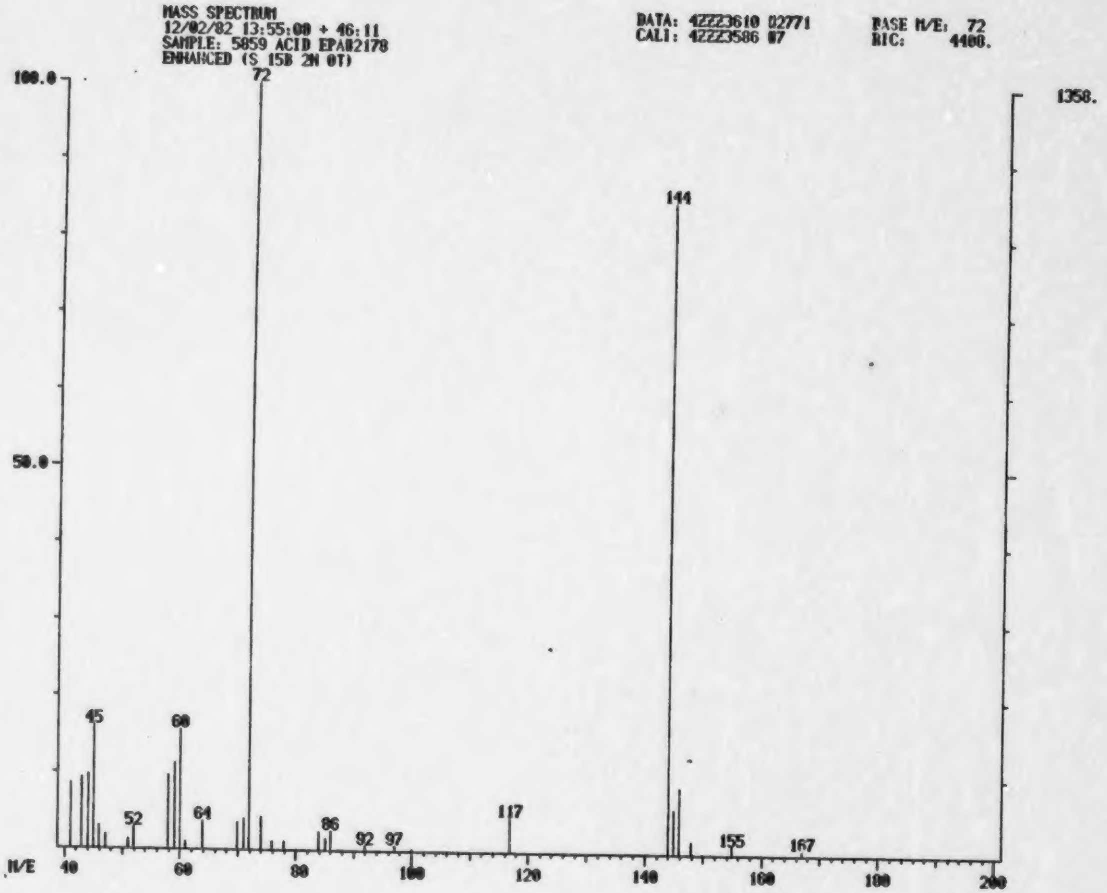


Figure B-95. Mass spectrum of EPA compound of interest, extract 5859 (acid), pesticides manufacturing industry, scan #2771.

Figure B-96. Conclusions

The presence of a high $(M + 2)^+$ peak suggests the presence of sulfur, as borne out by accurate mass measurement data (m/z 144, $C_4H_4N_2S_2$; error, -2.7 mmu). The spectrum is quite simple, suggesting a symmetry in the molecule. A small loss of 27 daltons is indicative of the presence of -CN in the molecule. The ion of mass 72 retains one sulfur atom. A probable structure is:

$$CN\,CH_2 - S - S - CH_2\,CN$$

No standard is available, however, to confirm this assignment.

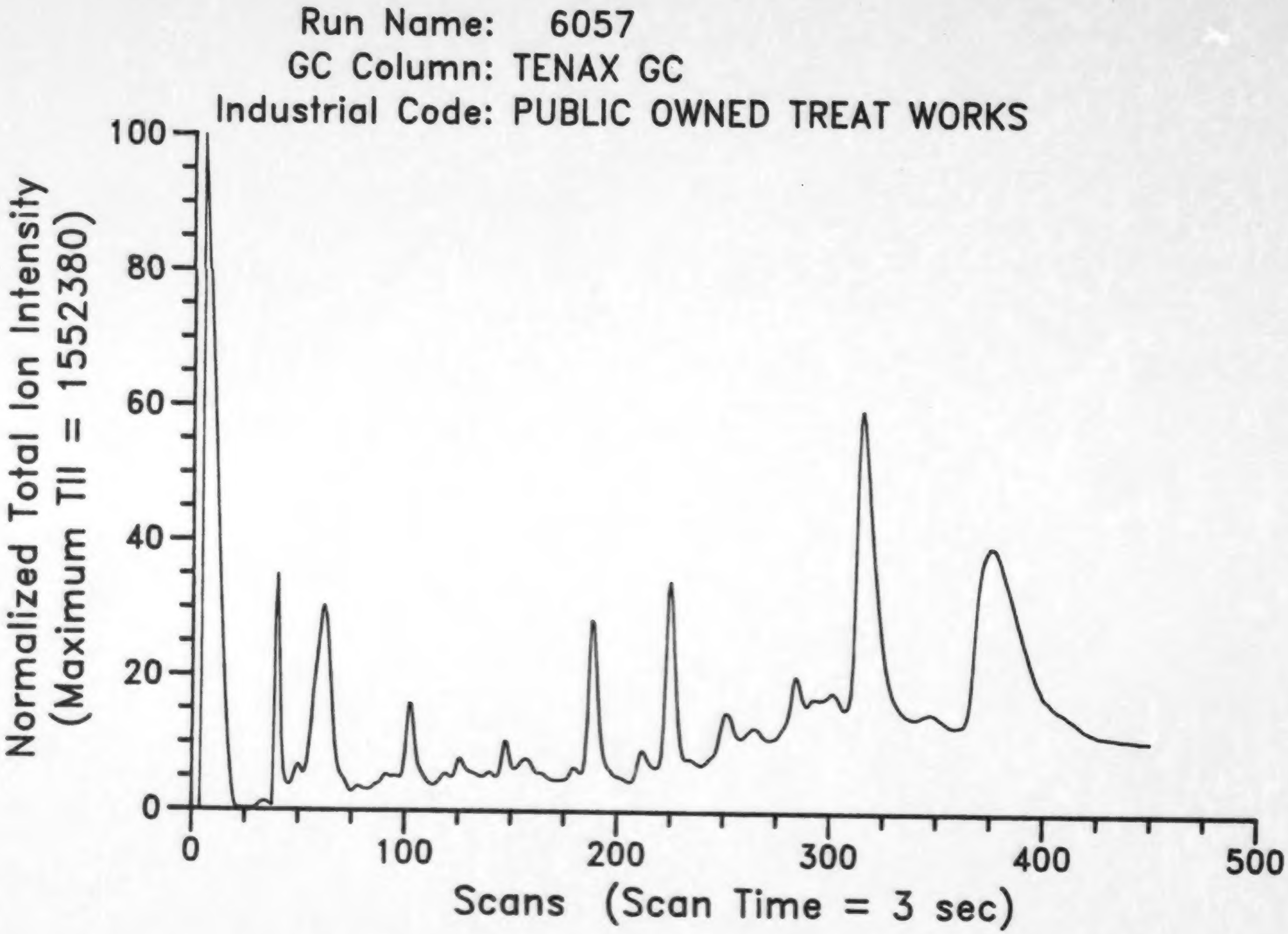


Figure B-97. Packed column chromatogram, extract 6057 (acid), publicy owned treatment works.

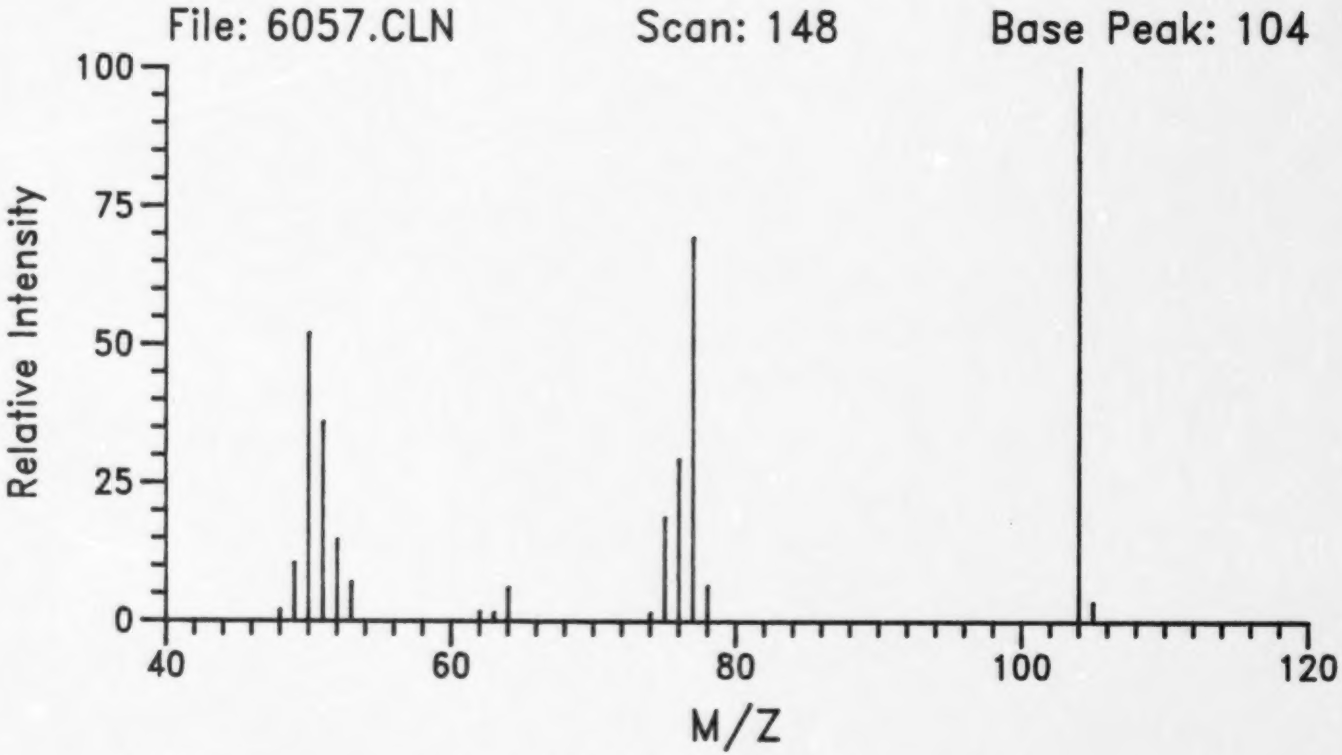


Figure B-98. Extract 6057 (acid), publicly owned treatment works. Frequency of occurrence: 6.

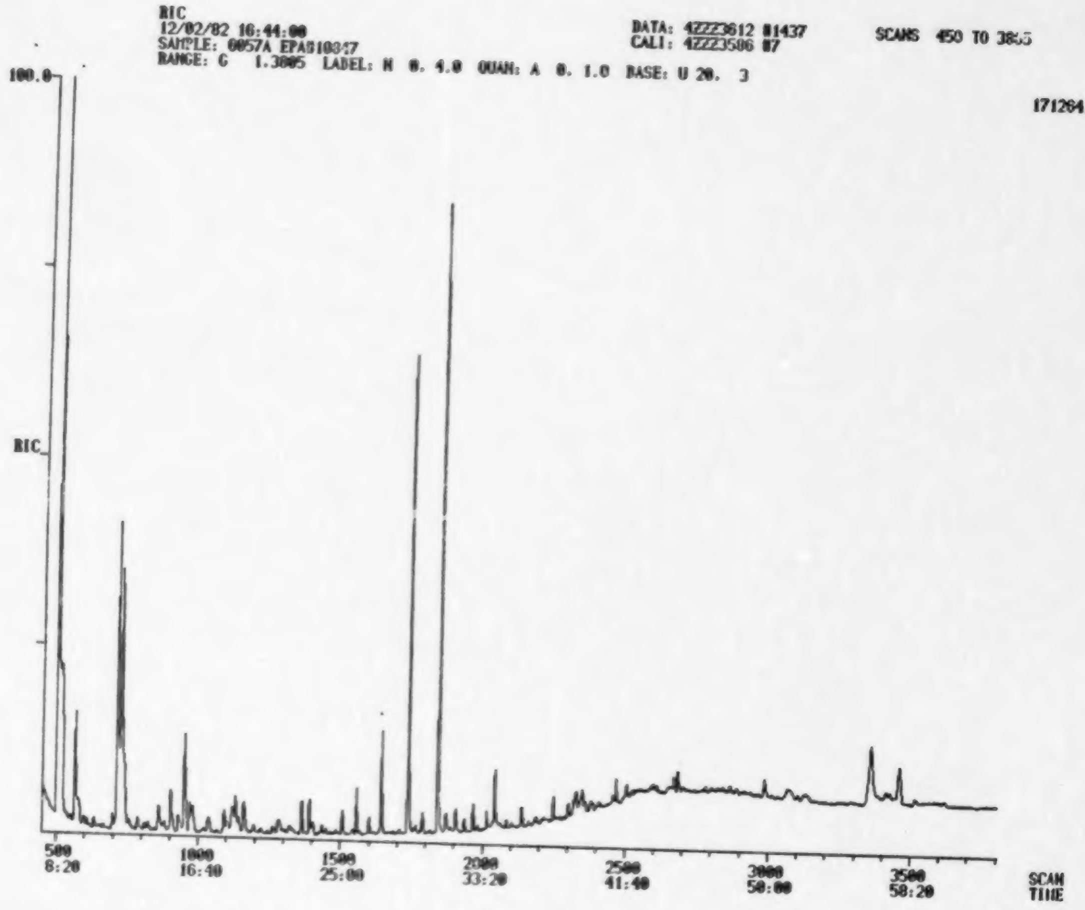


Figure B-99. Capillary chromatogram, extract 6057 (acid), publicly owned treatment works.

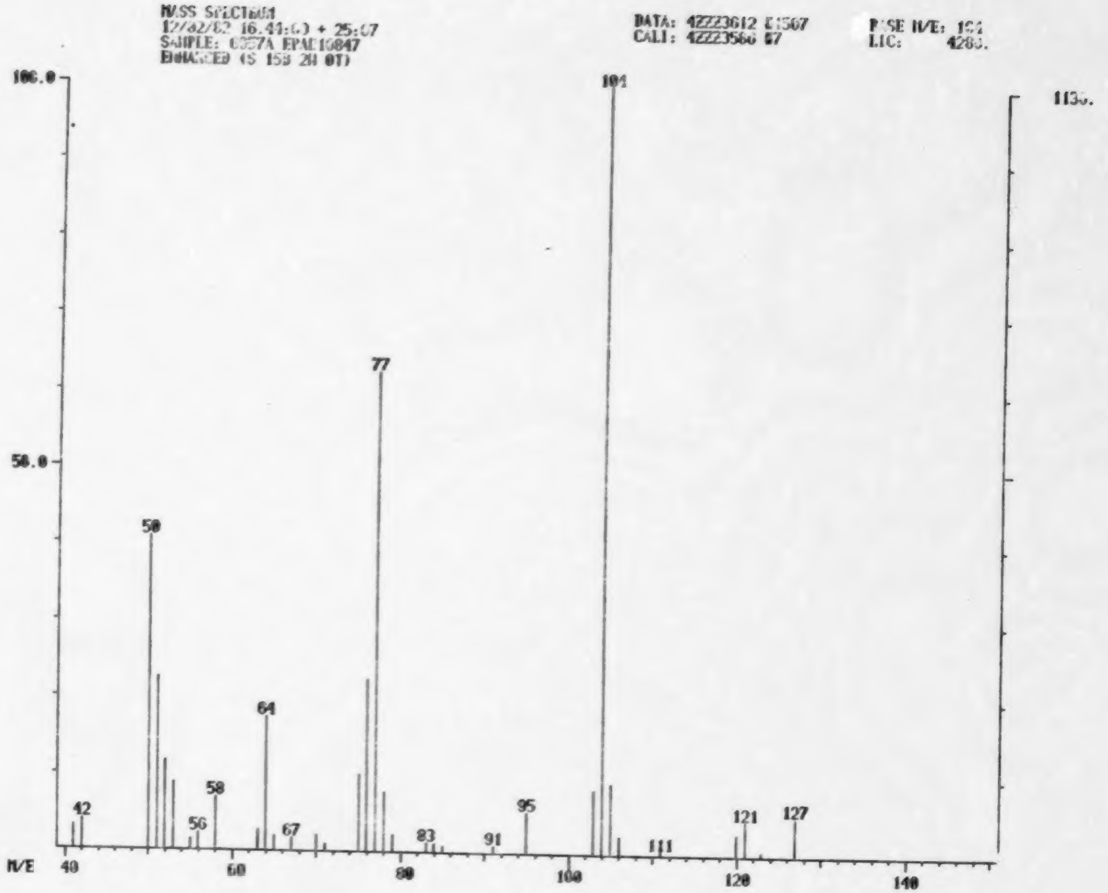


Figure B-100. Mass spectrum of EPA compound of interest, extract 6057 (acid), publicly owned treatment works, one isomer, scan #1507.

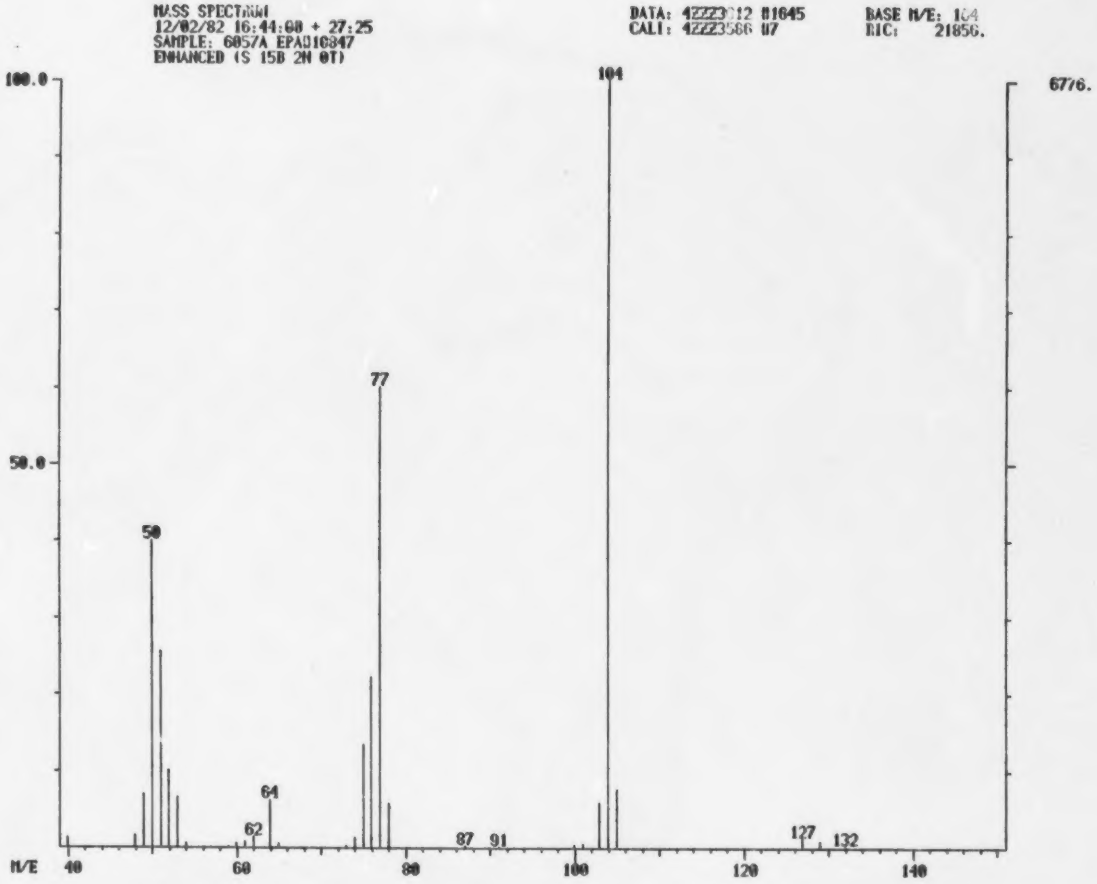


Figure B-101. Mass spectrum of EPA compound of interest, extract 6057 (acid), publicly owned treatment works, one isomer, scan #1645.

Figure B-102. Conclusions

Accurate mass measurement yields two possibilities for molecular composition, $C_4H_8O_3$ (error, 3.3 mmu) and $C_6H_4N_2$ (error, -6.6 mmu). The presence of such an intense peak at $\underline{m}/\underline{z}$ 77 (a loss of 27 mass units) is strongly suggestive of the presence of nitrogen, especially a -CN group. A structure such as a cyanopyridine could be drawn for the molecule and the mass spectra correspond quite closely, but a compound of this type should be found in an acid fraction. Perhaps if the compound of interest is indeed a cyanopyridine, it is being formed from another component of the extract, or results from carryover from a large quantity of this compound in the base/neutral fraction. It would be useful to examine the base/neutral fraction of this industrial effluent.

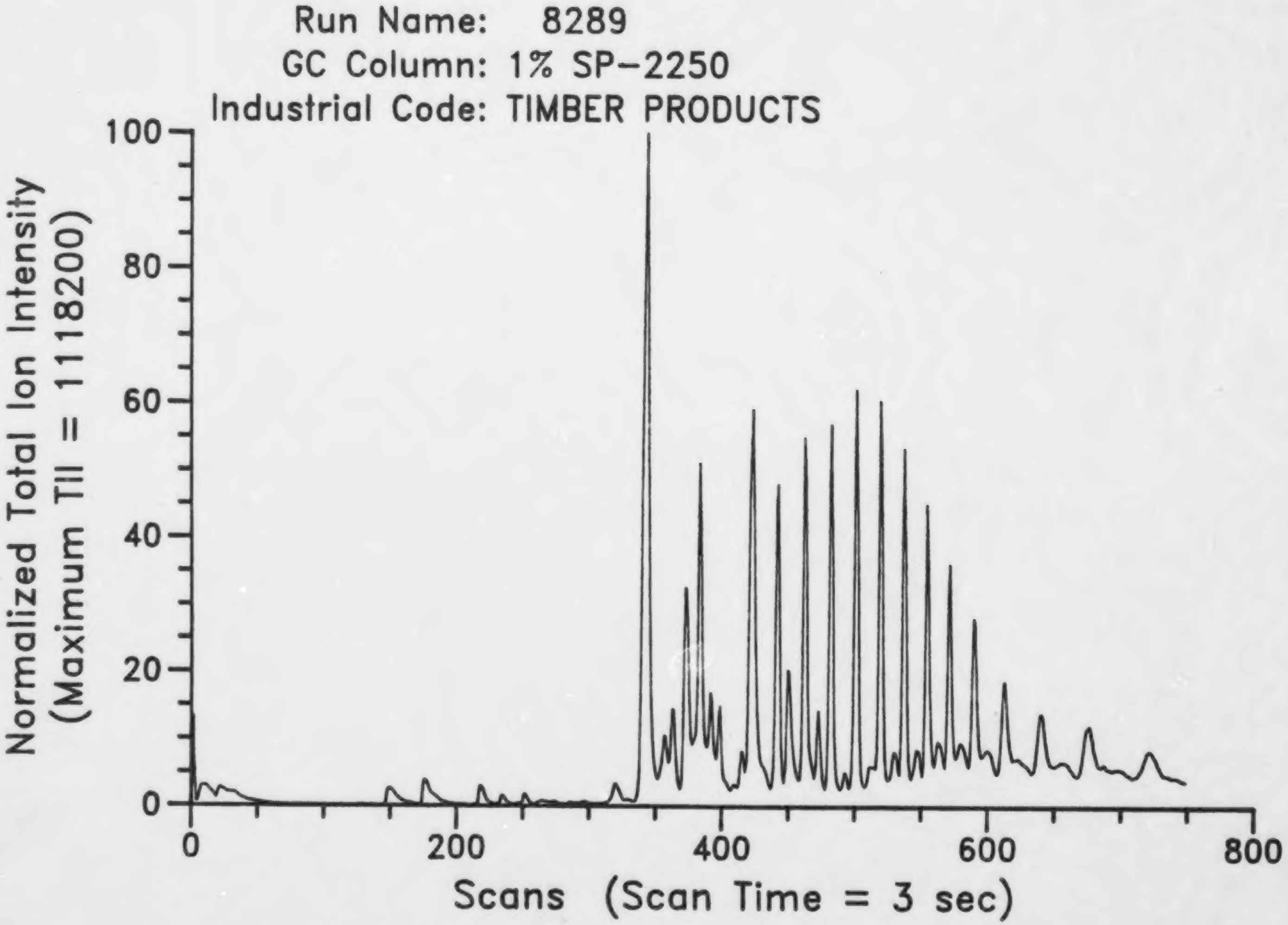


Figure B-103. Packed column chromatogram, extract 8289 (base/neutral), timber products industry.

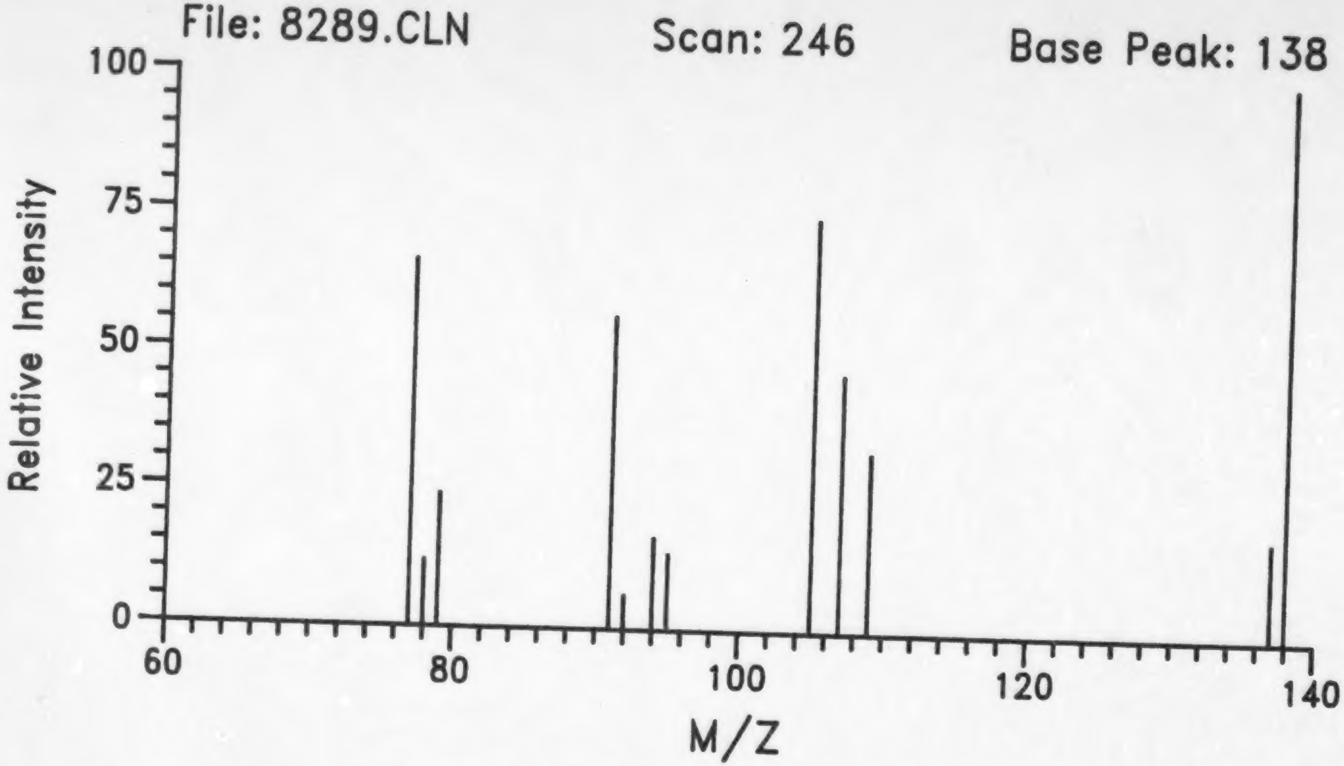


Figure B-104. Extract 8289 (base/neutral), timber products industry. Frequency of occurrence: 5.

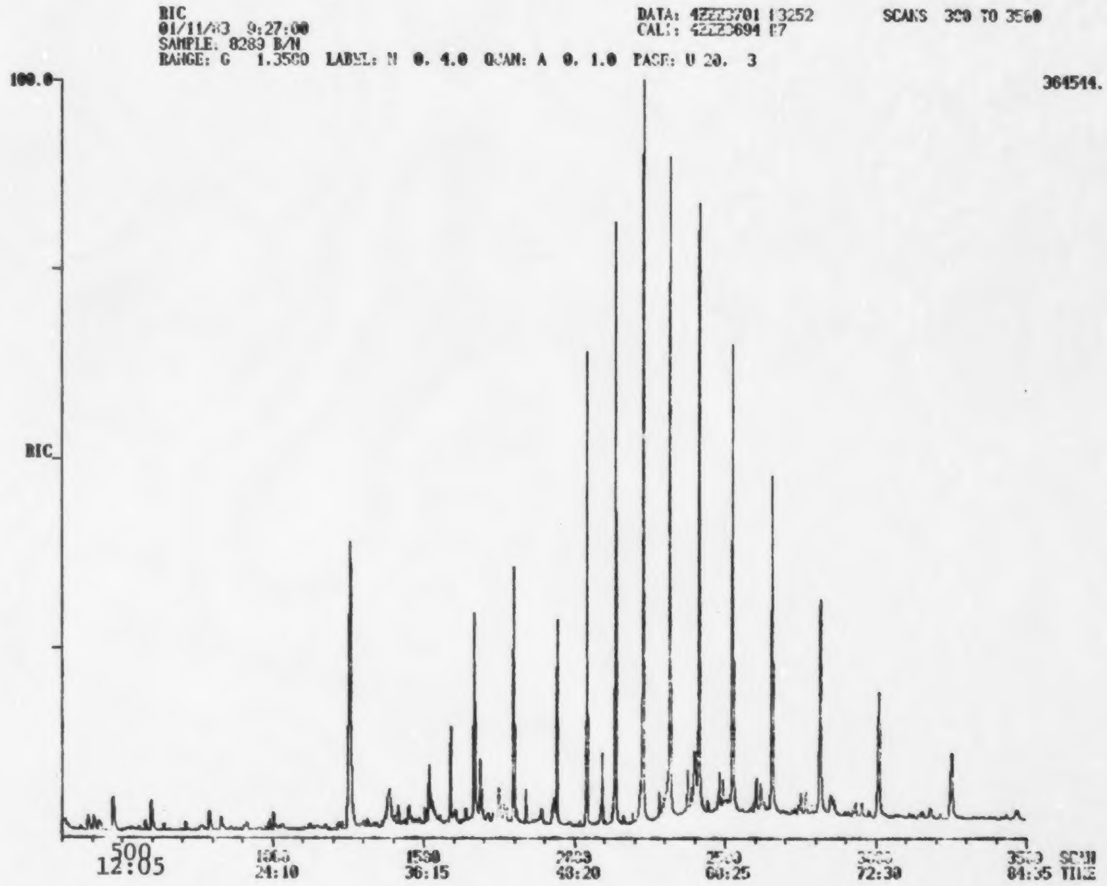


Figure B-105. Capillary chromatogram, extract 8289 (base/neutral), timber products industry.

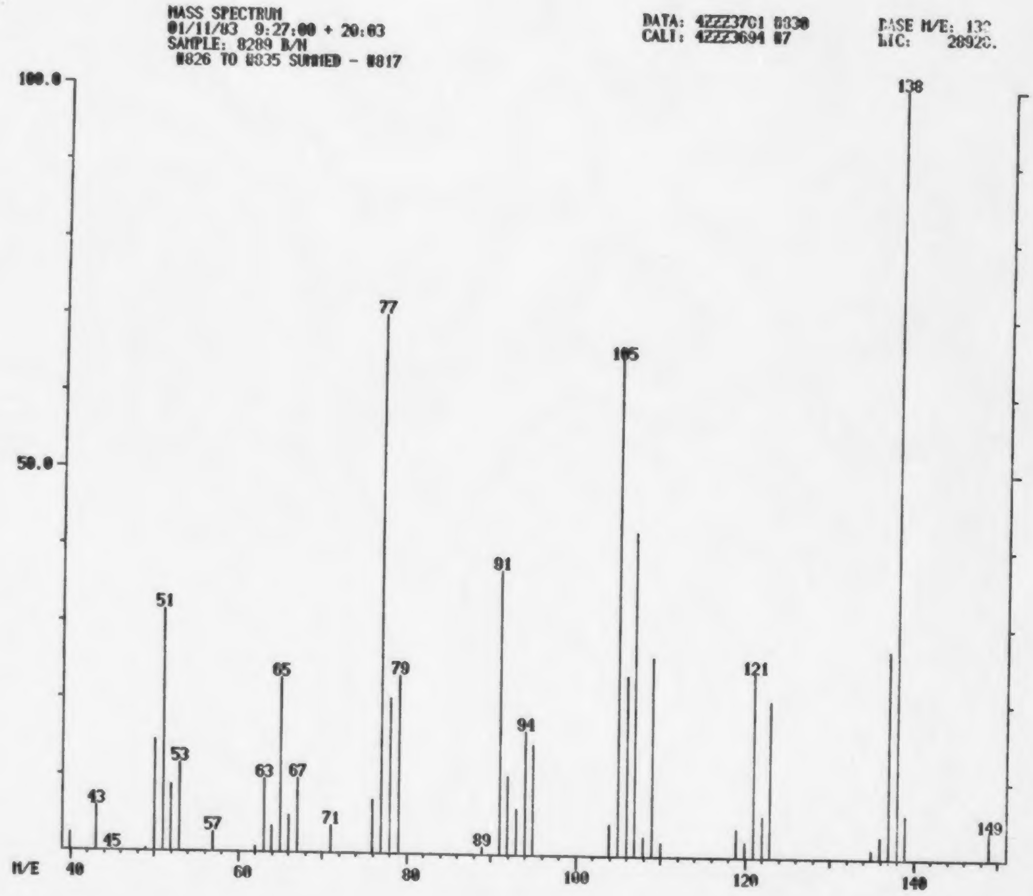
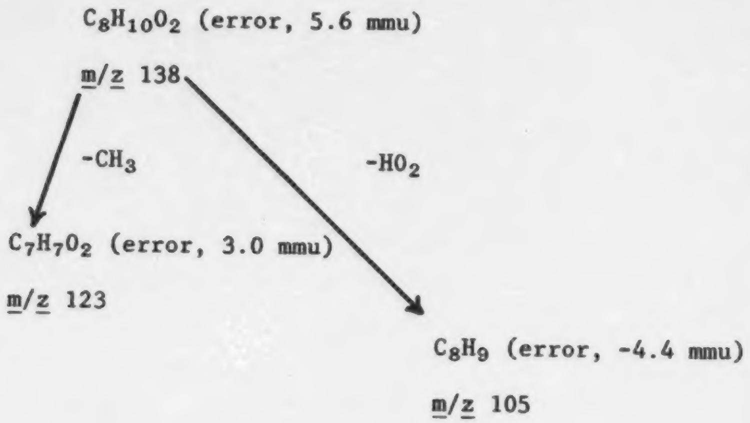


Figure B-106. Mass spectrum of EPA compound of interest, extract 8289 (base/neutral), timber products industry, scan #830.

Figure B-107. Conclusions

Accurate mass measurement produces only one possibility for three of the major ions of the mass spectrum.


$C_8H_{10}O_2$ (error, 5.6 mmu)
m/z 138
$-CH_3$
$-HO_2$
$C_7H_7O_2$ (error, 3.0 mmu)
m/z 123
C_8H_9 (error, -4.4 mmu)
m/z 105

The presence of the hydrocarbon ion at mass 105 and the lower mass ions at 77, 65, and 51 are indicative of the presence of an aromatic ring with two carbon atoms attached to it. The carbon atoms may be attached to each other (*i.e.*, an ethyl group) or individually. The compound mass spectrum shows an ion at mass 123, showing that the compound can lose 17 mass units (an -OH group) under electron bombardment. If the loss of HO_2 is indeed correct, this grouping would indicate that the two oxygen atoms must be bonded together in the original molecule. This postulation is difficult to support, since no reference spectra are available and the stability of such a compound in the original water would be questionable. One cannot exclude the possibility that, if the identification as a peroxide has any validity, this type of compound could be formed in the course of the analytical process.

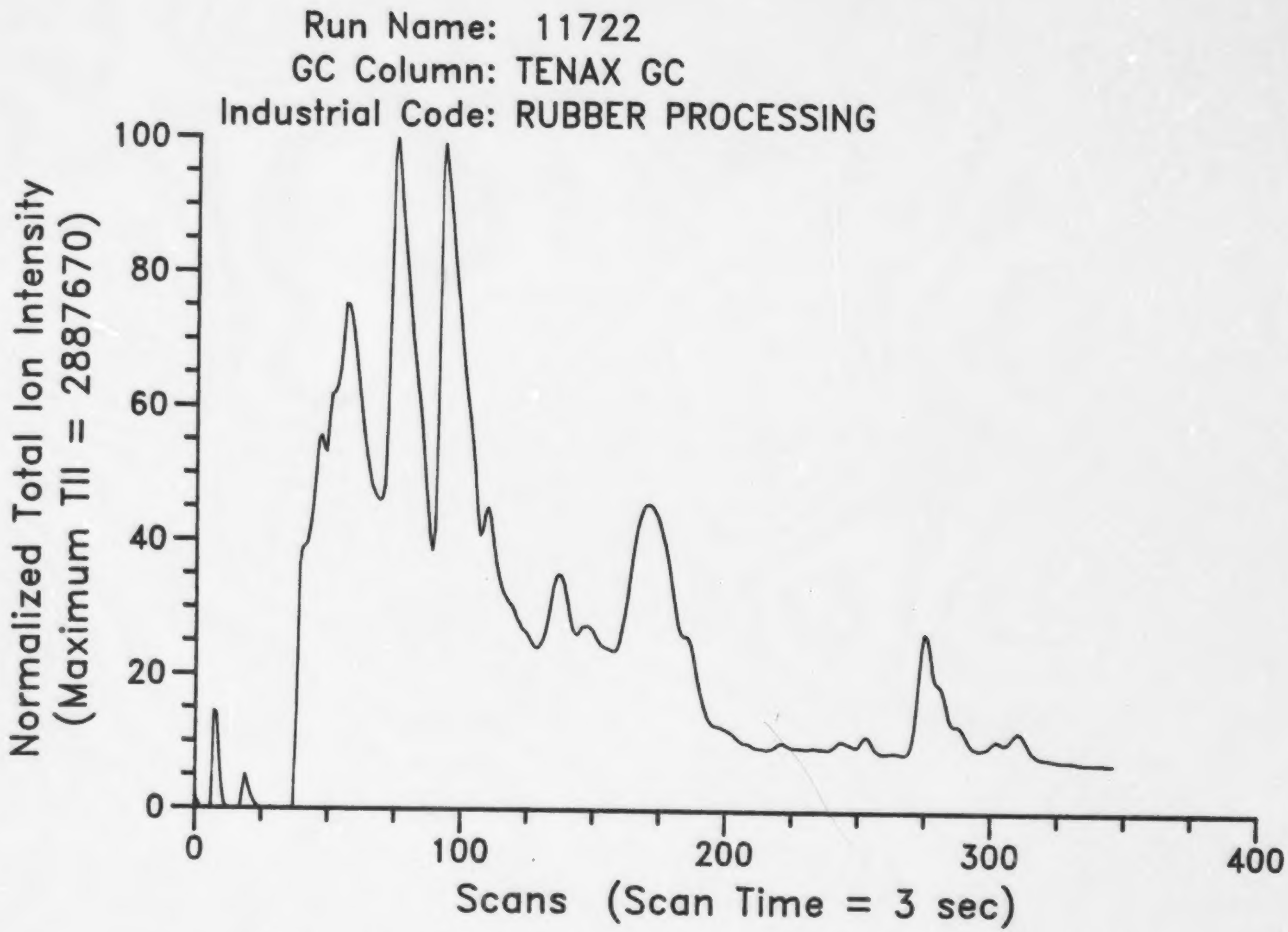


Figure B-108. Packed column chromatogram, extract 11722 (acid), rubber processing industry.

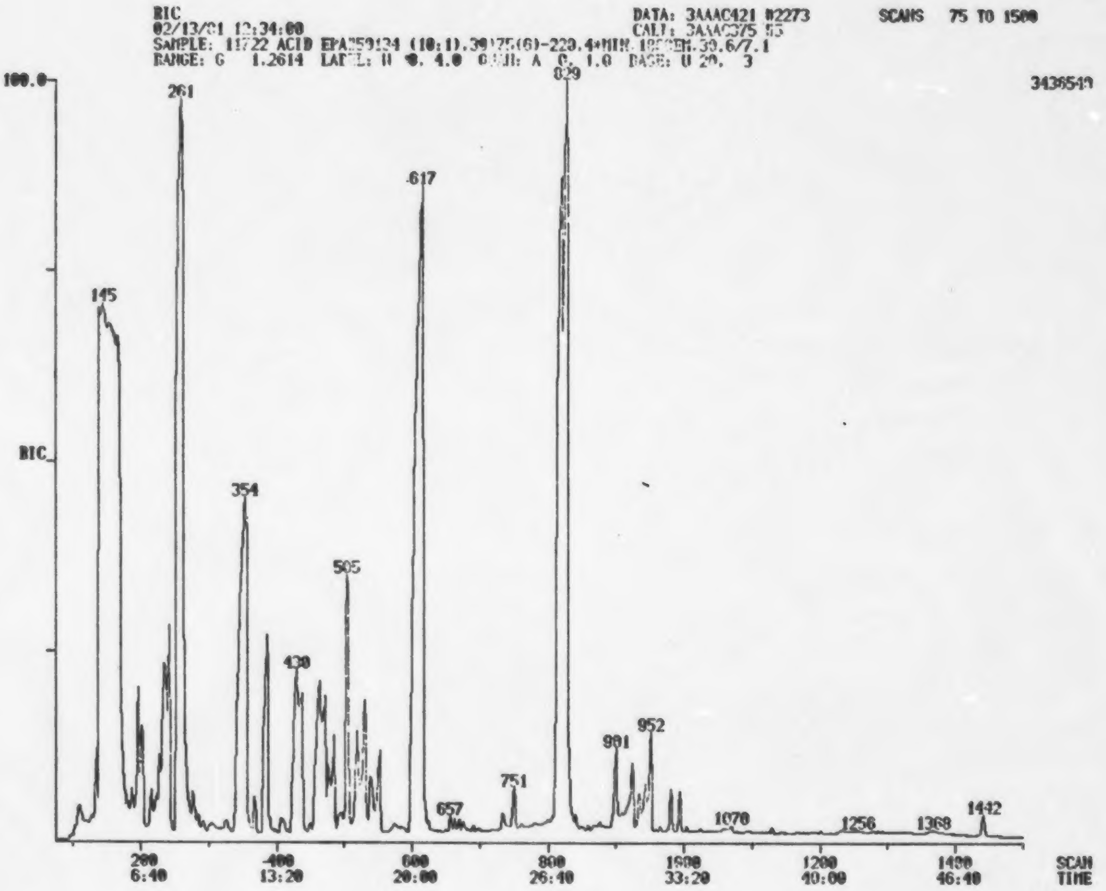


Figure B-109. Capillary chromatogram, extract 11722 (acid), rubber processing industry.

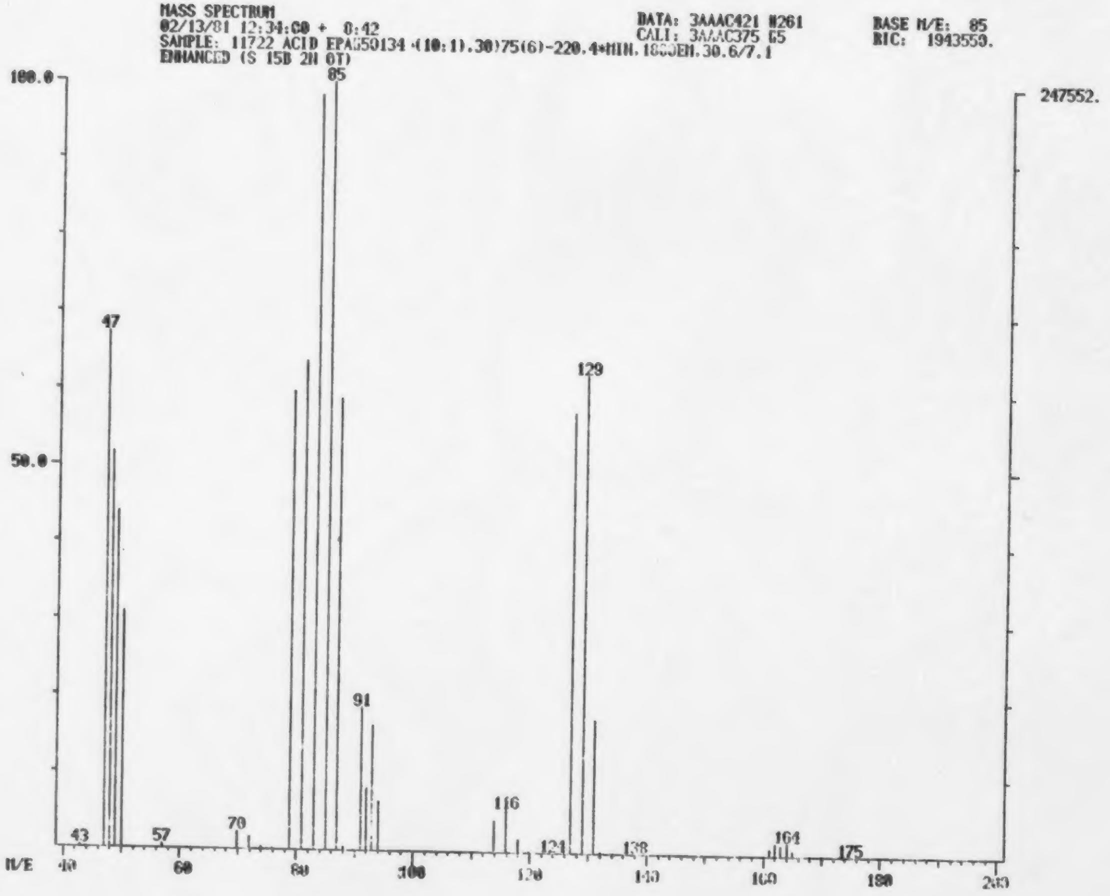


Figure B-110. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #261.

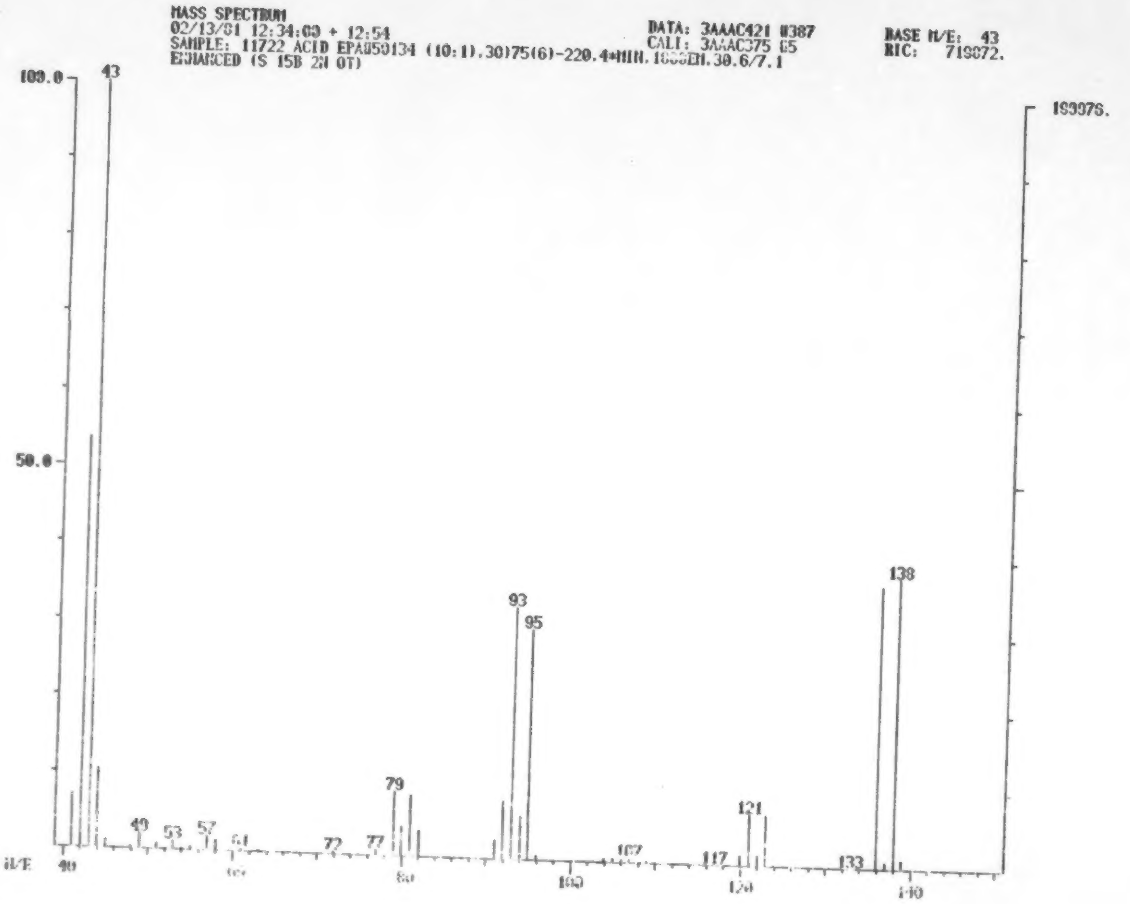


Figure B-111. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #387.

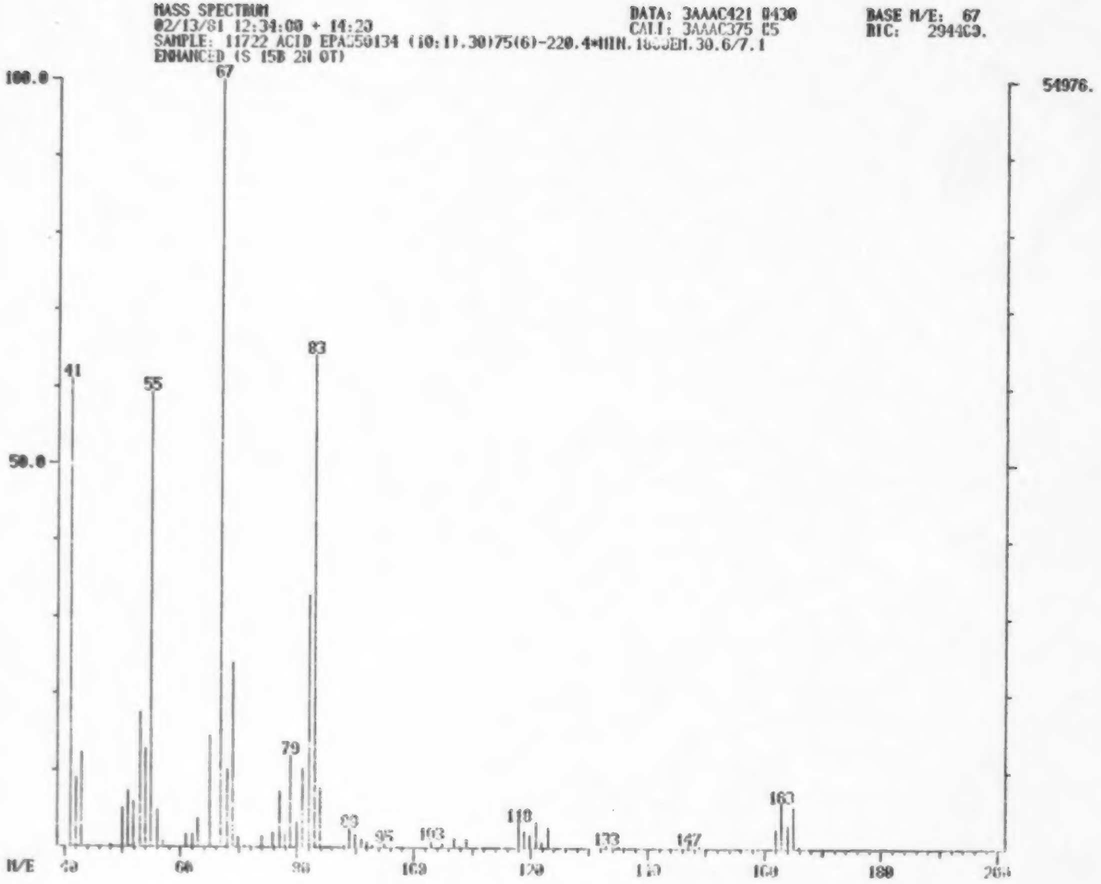


Figure B-112. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #430.

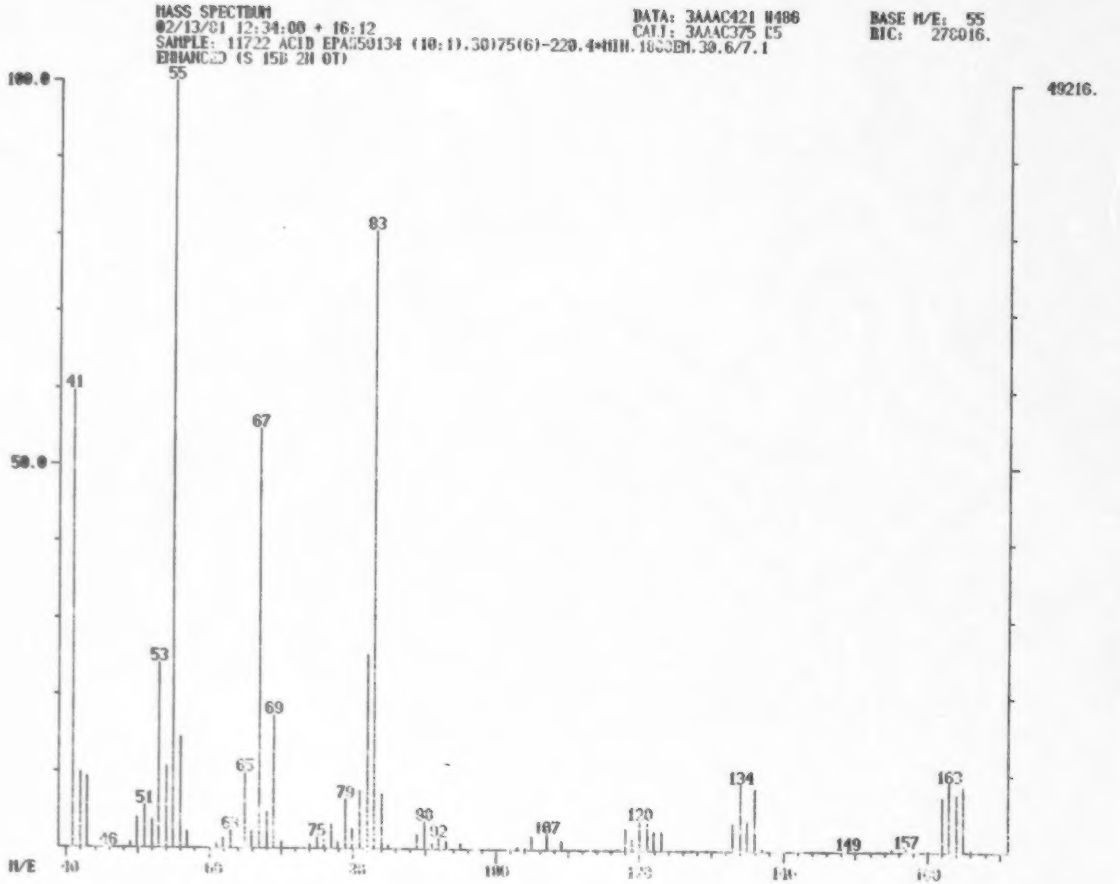


Figure B-113. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #486.

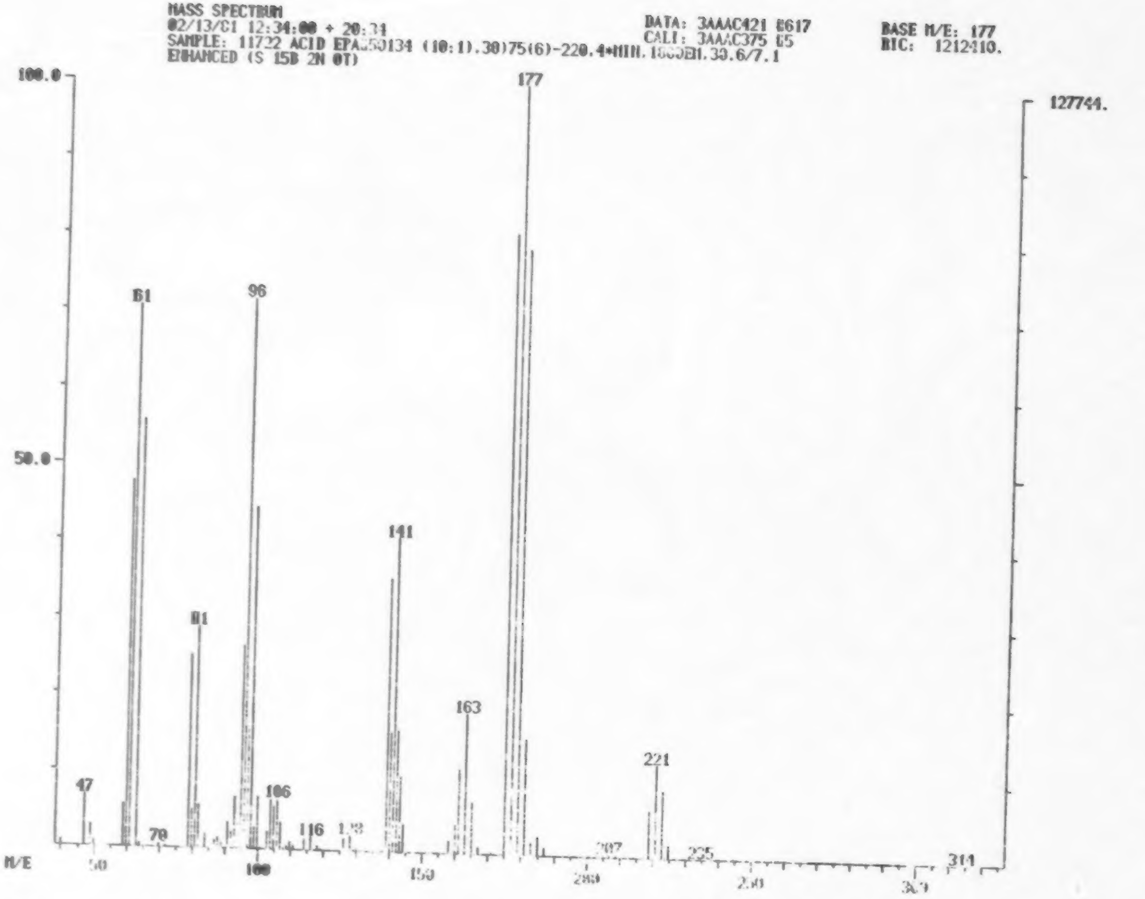


Figure B-114. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #617.

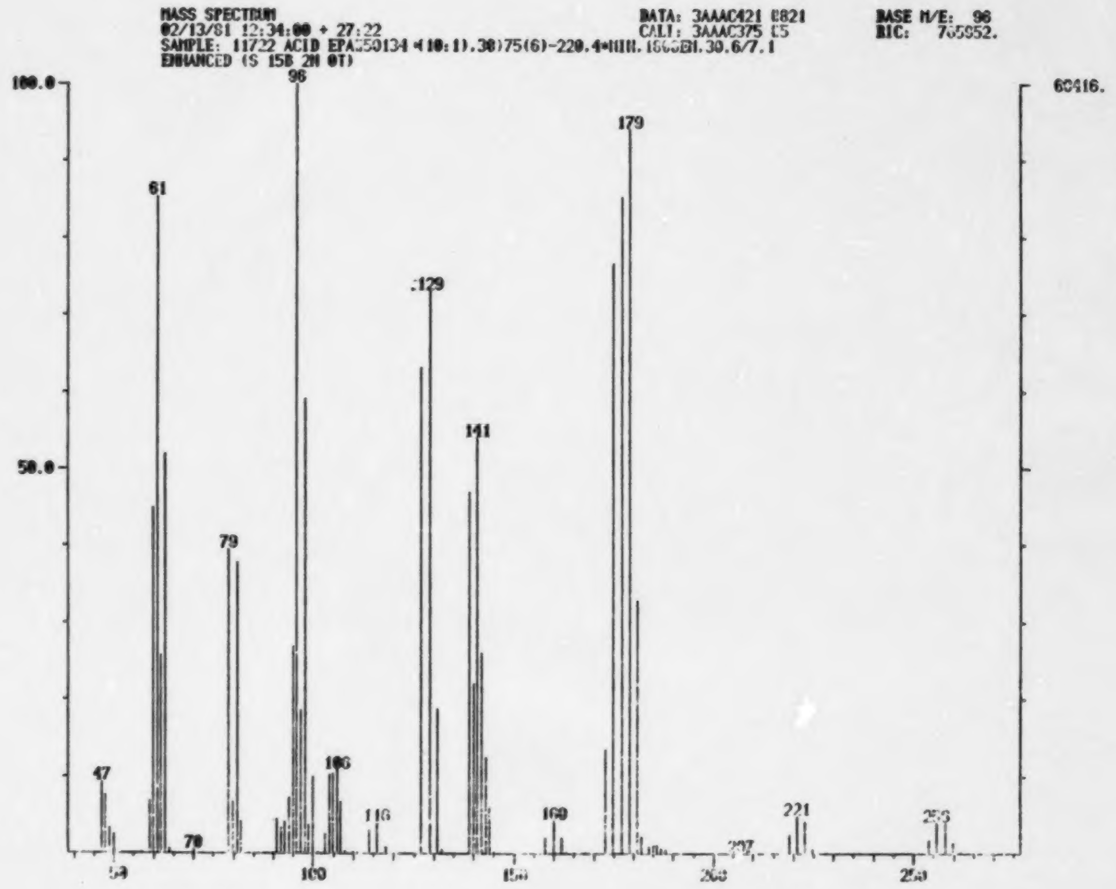


Figure B-115. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #821.

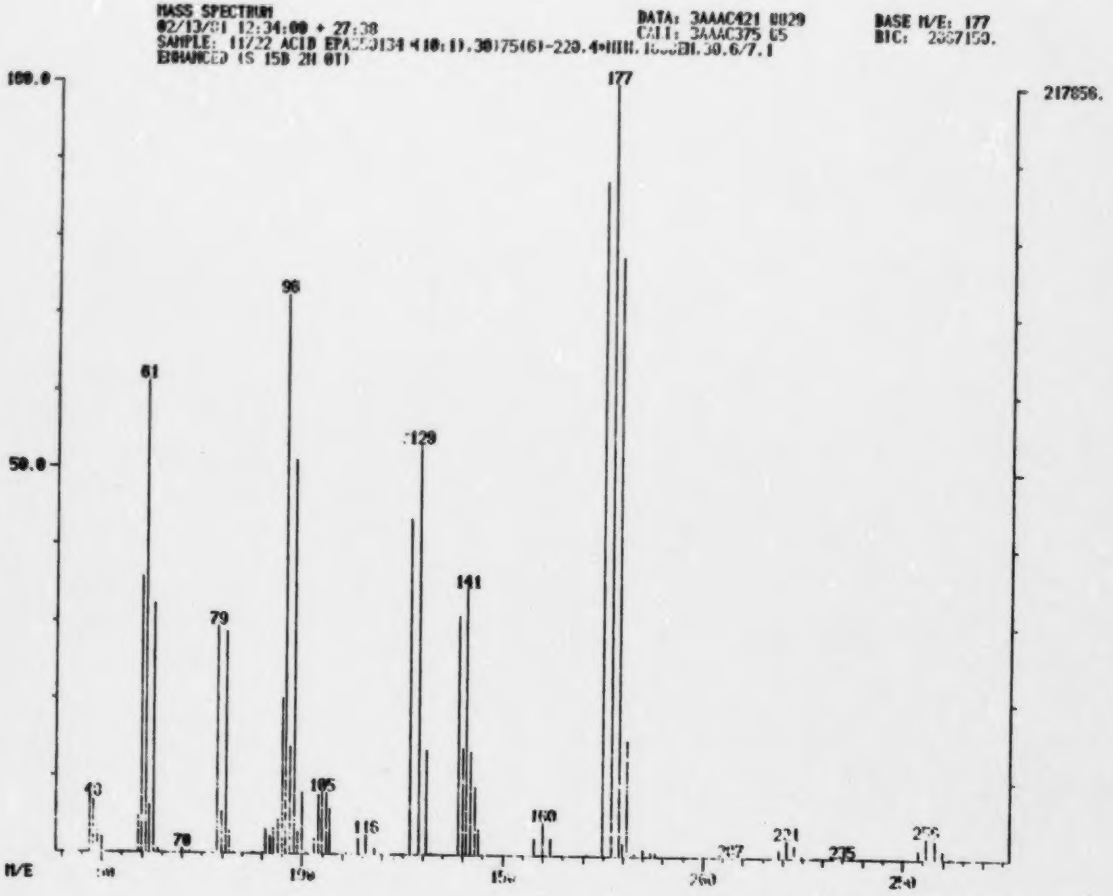


Figure B-116. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #829.

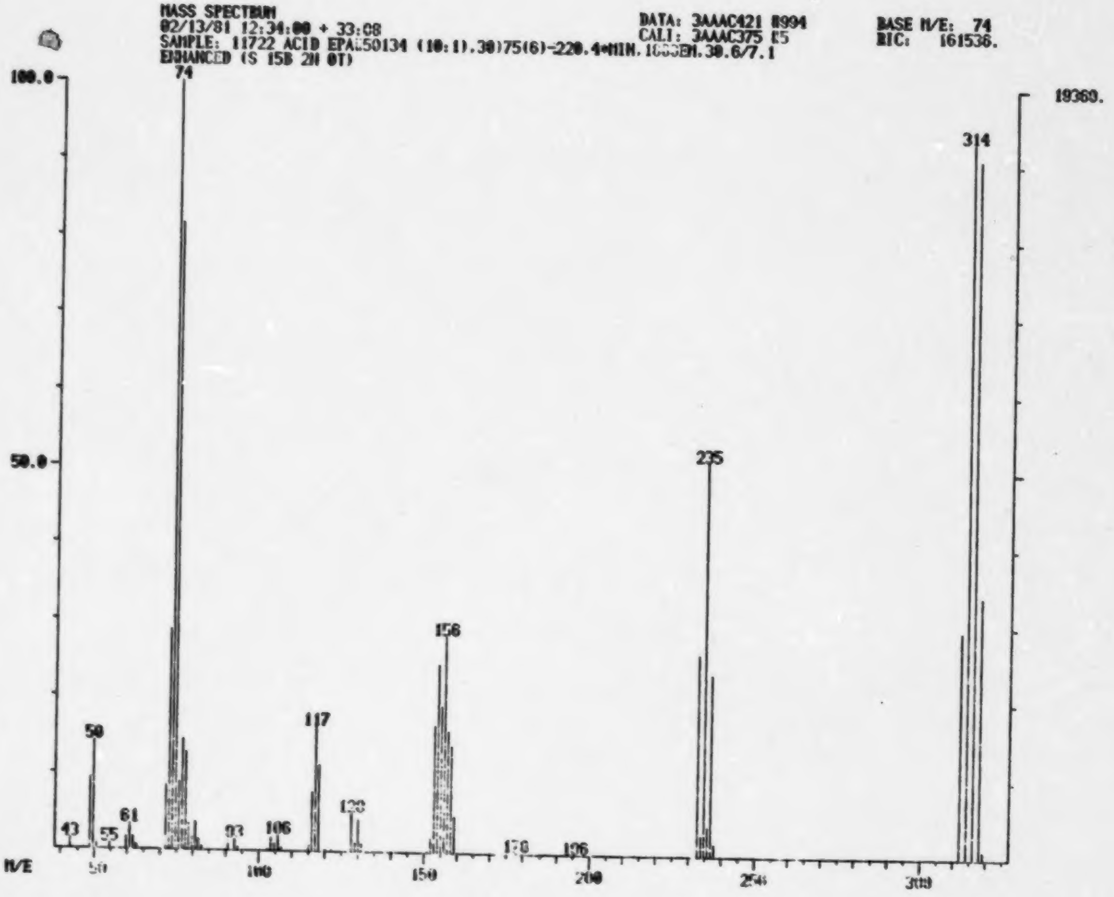


Figure B-117. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #994.

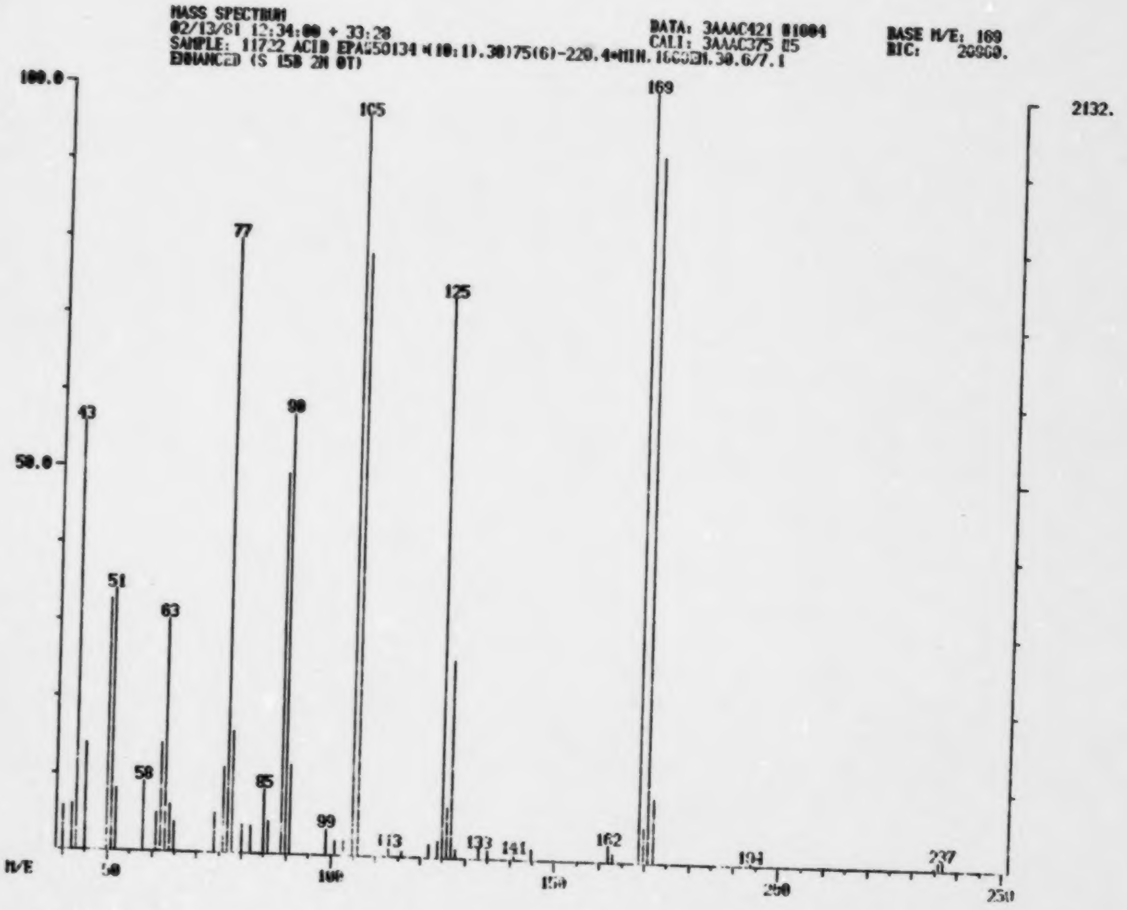


Figure B-118. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1604.

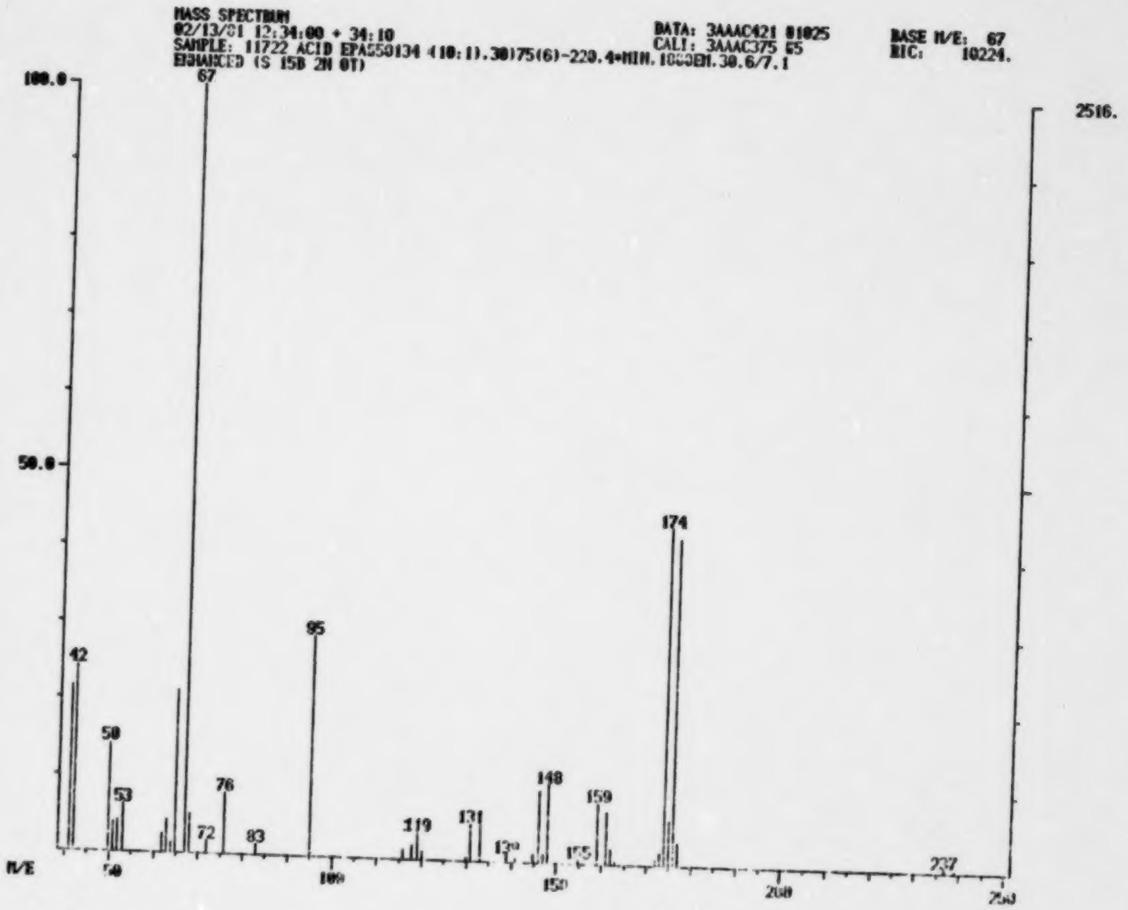


Figure B-119. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1025.

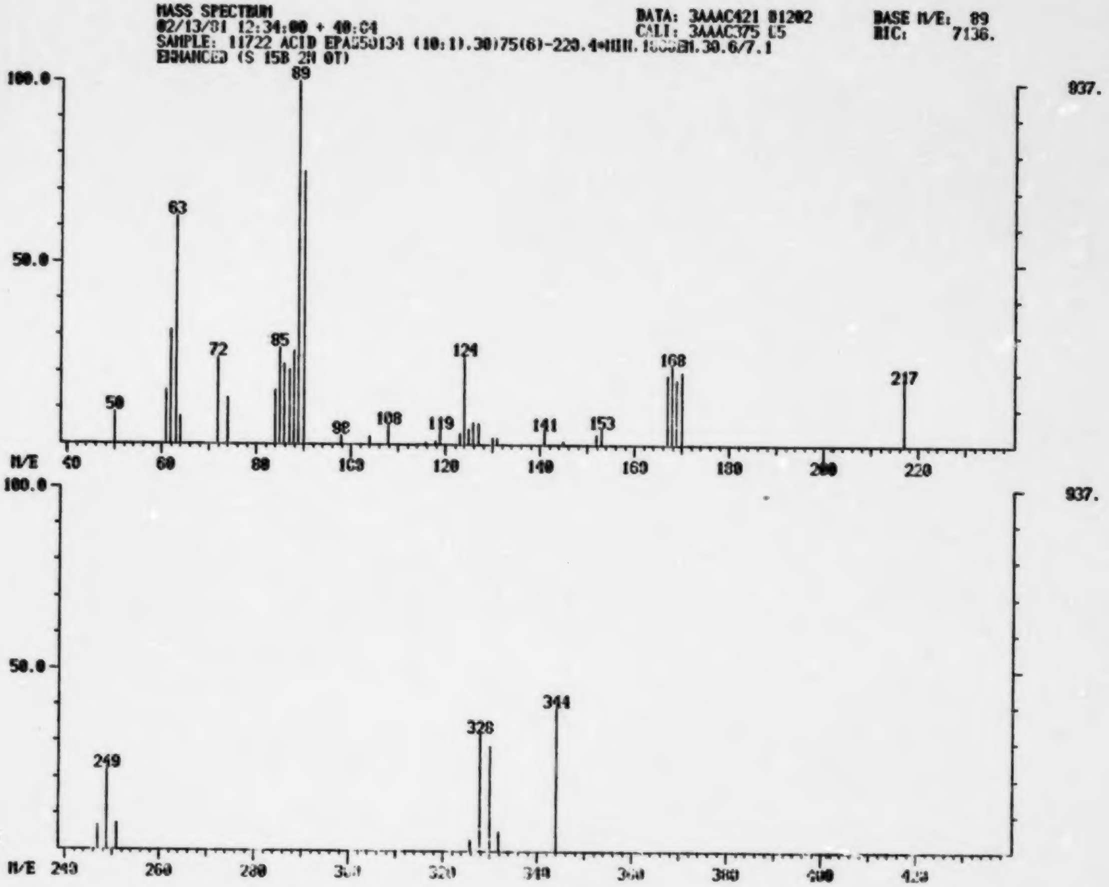


Figure B-120. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1202.

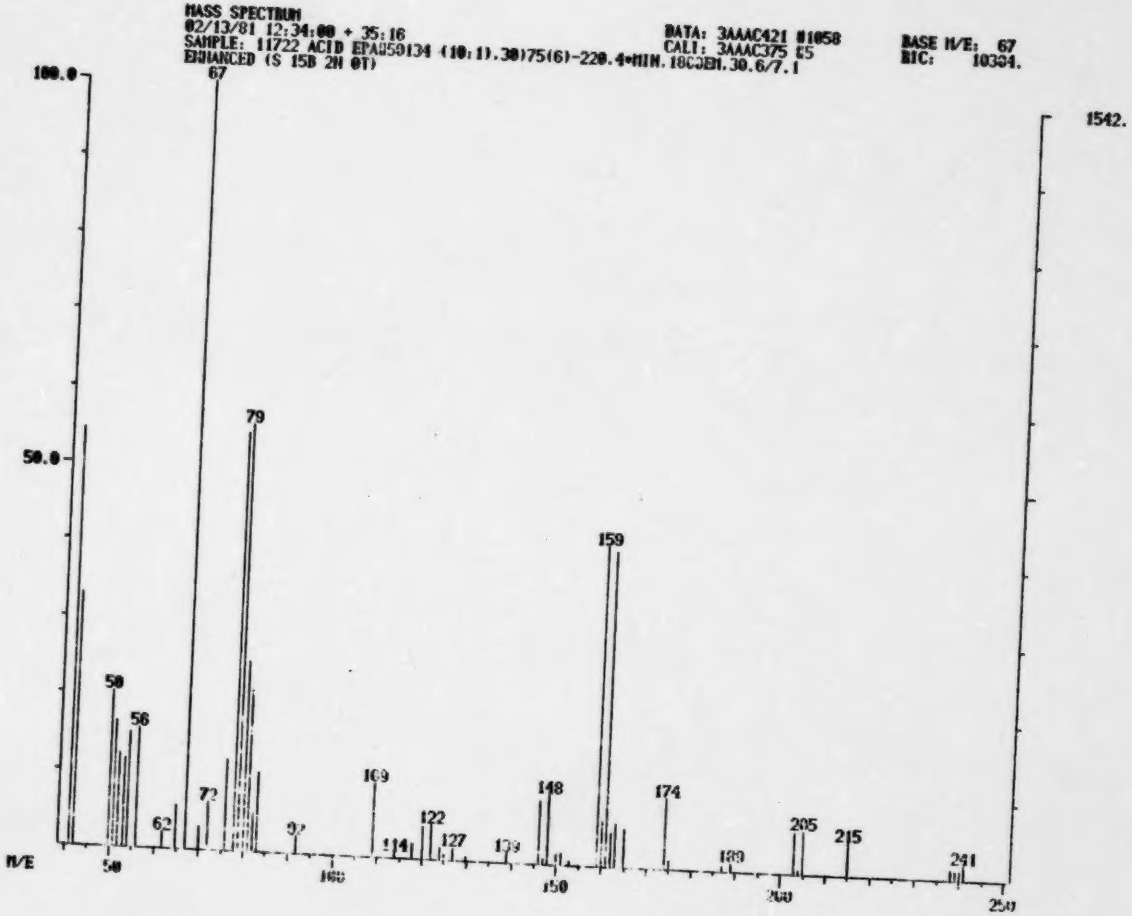


Figure B-121. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1058.

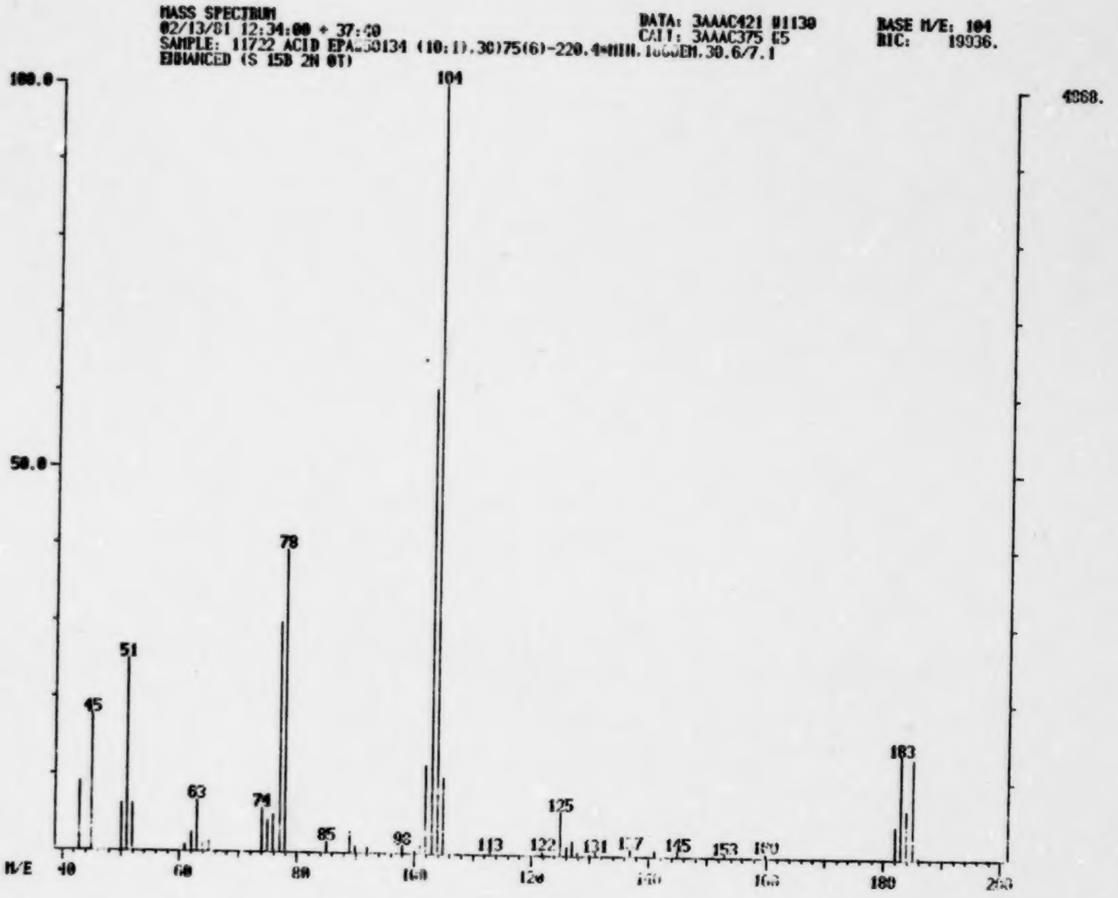


Figure B-122. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1130.

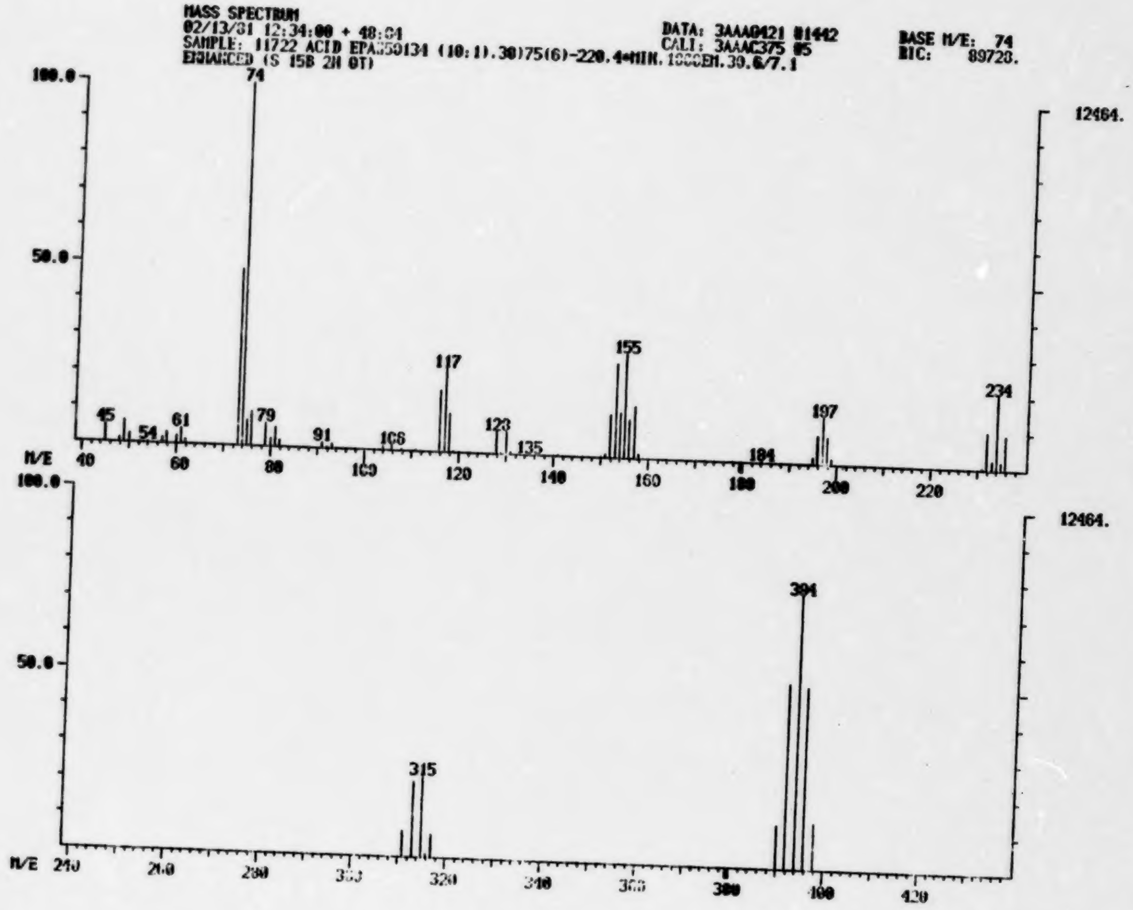


Figure B-123. Mass spectrum of unidentified brominated constituent, extract 11722 (acid), rubber processing industry, scan #1442.

Figure B-124. Tentatively Identified Compounds

Scan	Compound
135	Vinylidene chloride
368	Dibromochloromethane
505	Bromoform
734	Dibromocyclohexane isomer
751	Dibromocyclohexane isomer
901	Dibromocyclohexane isomer

NOTE: Bromobenzenes, up to Br_4, were observed under negative ion chemical ionization conditions.

Figure B-125. Conclusions

There are obviously many brominated compounds present in this extract. The compounds are readily seen as brominated but it is as readily seen that the ion of highest mass observed under electron ionization conditions is not a molecular species. Chemical ionization mass spectrometry, both positive and negative modes, was employed in an effort to obtain more information as to the nature of the molecular species. Several pieces of information were obtained by chemical ionization: (1) use of chemical ionization, either positive or negative mode, frequently shows halogenated ions of higher mass than the species observed under electron ionization. However, these ions of higher mass (and possibly higher bromine number) do not represent molecular ion species, either; (2) use of negative ion chemical ionization makes several bromobenzenes (up to four bromine atoms) observable, where these compounds could not be observed under electron ionization conditions. Given the types of compounds which could be tentatively identified, many of the unidentified compounds are probably brominated cyclohexanes and cyclohexenes. Analysis of standards would be essential in obtaining reference spectra and establishing relative retention times, but no standards for these classes of compounds are commercially available.

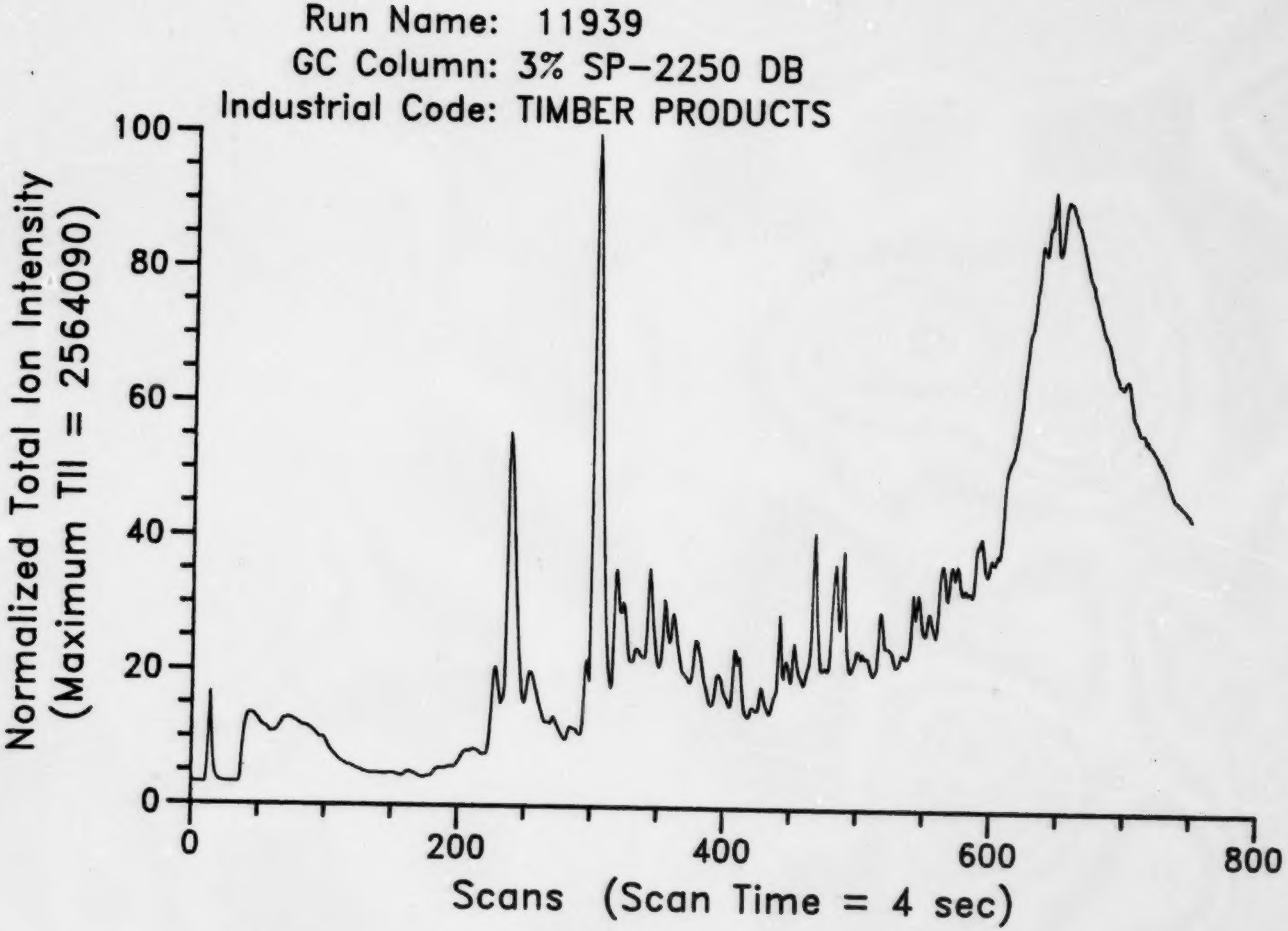


Figure B-126. Packed column chromatogram, extract 11939 (base/neutral), timber products industry.

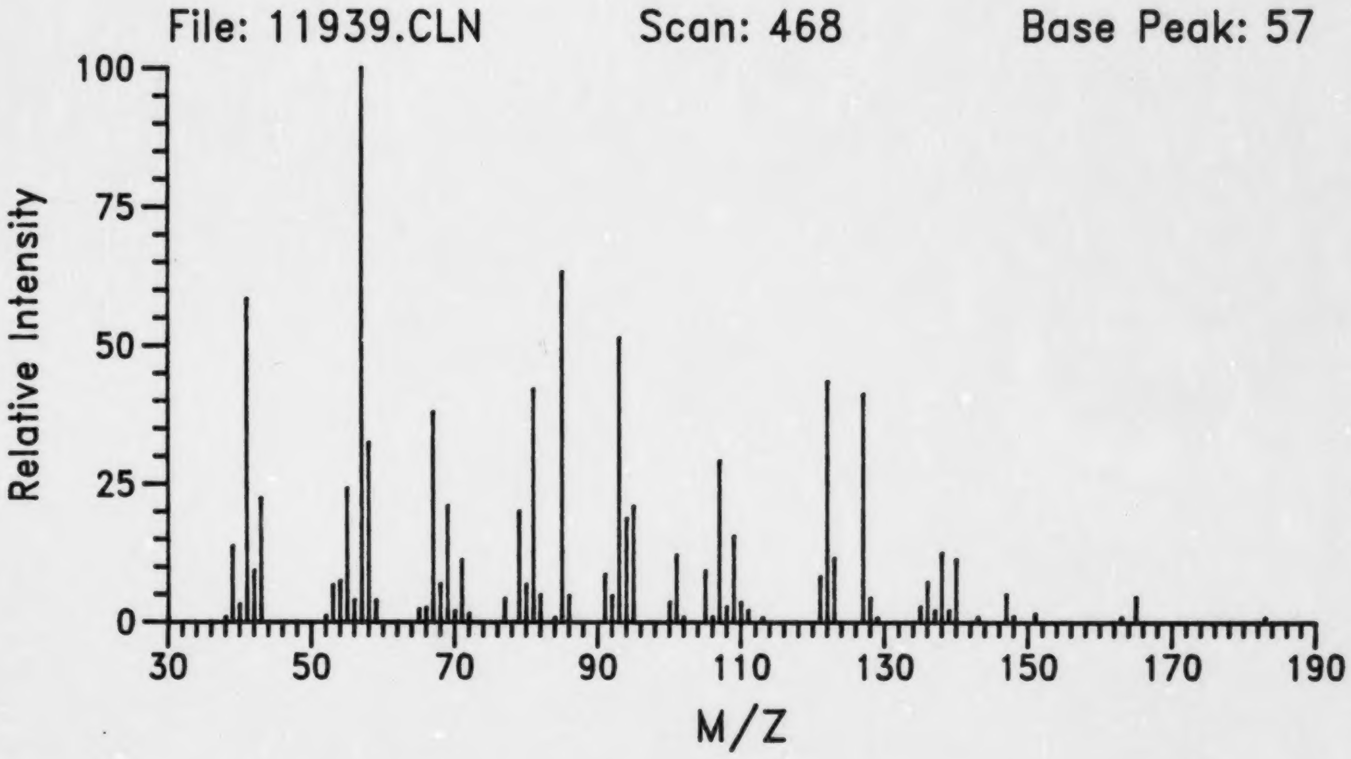


Figure B-127. Extract 11939 (base/neutral), timber products industry. Frequency of occurrence: 5.

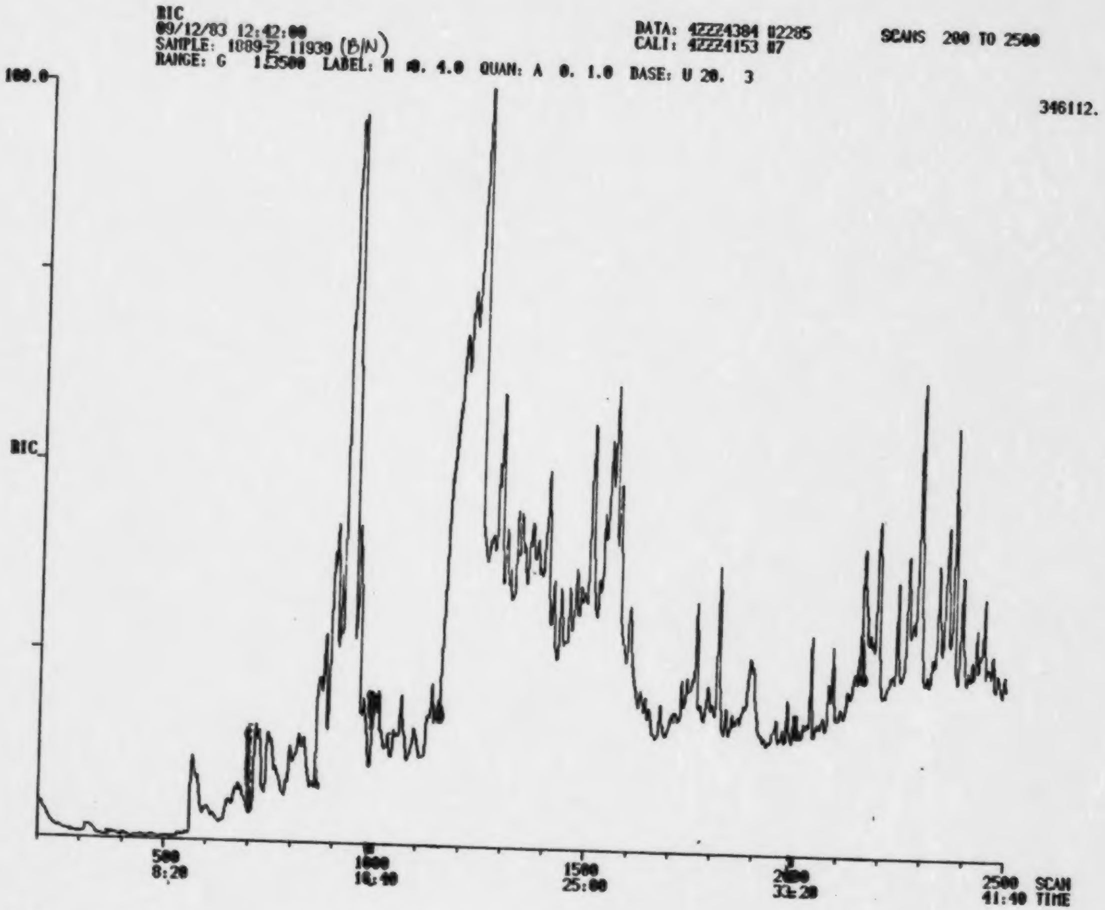


Figure B-128. Capillary chromatogram, extract 11939 (base/neutral), timber products indusry.

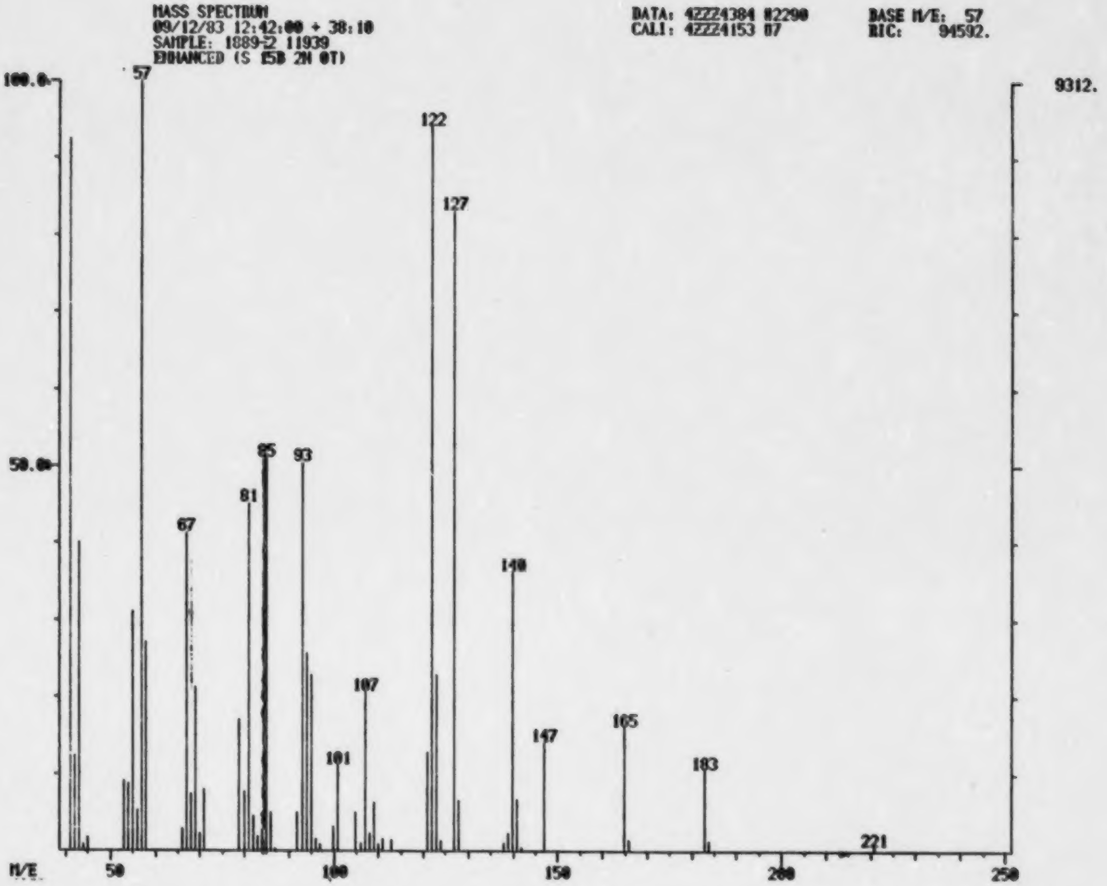


Figure B-129. EPA compound of interest, extract 11939 (base/neutral), timber products industry.

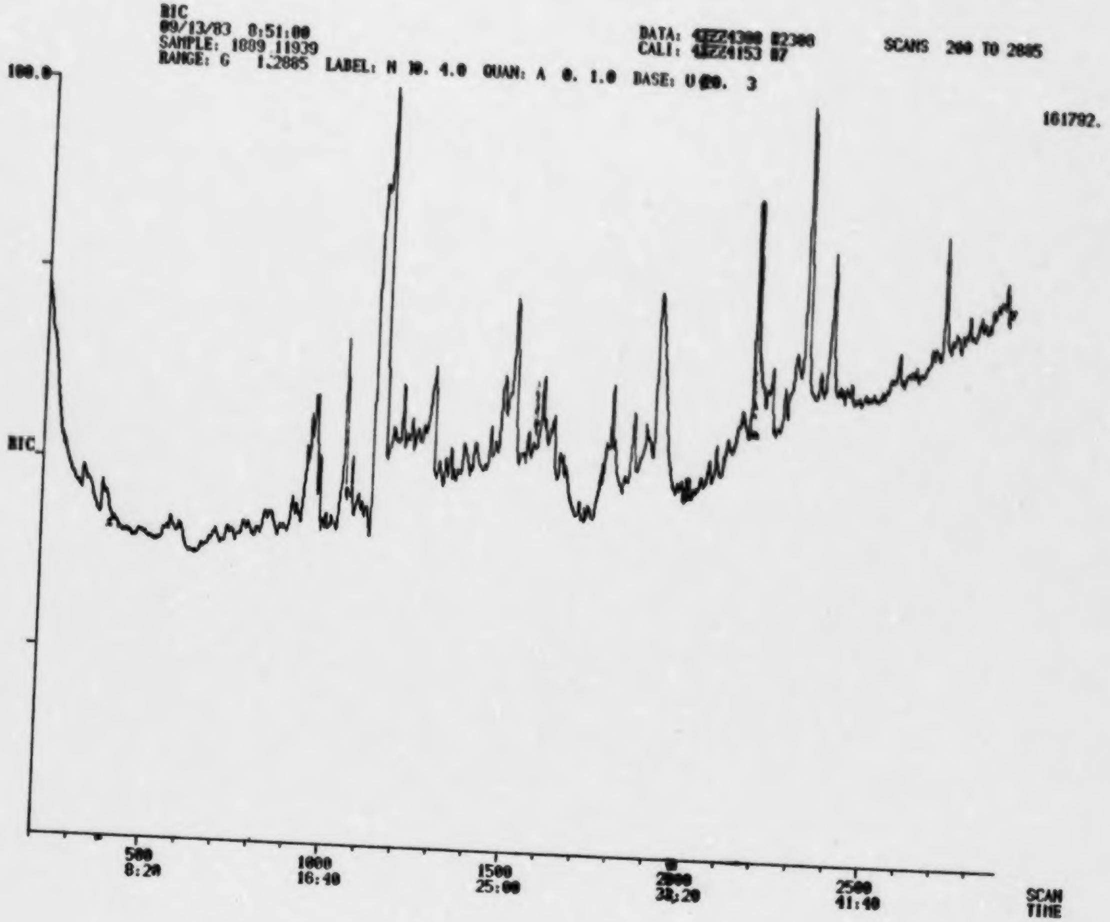


Figure B-130. Capillary chromatogram (chemical ionization mass spectrometry), extract 11939 (base/neutral), timber products industry. Note lack of correspondence to EI profile.

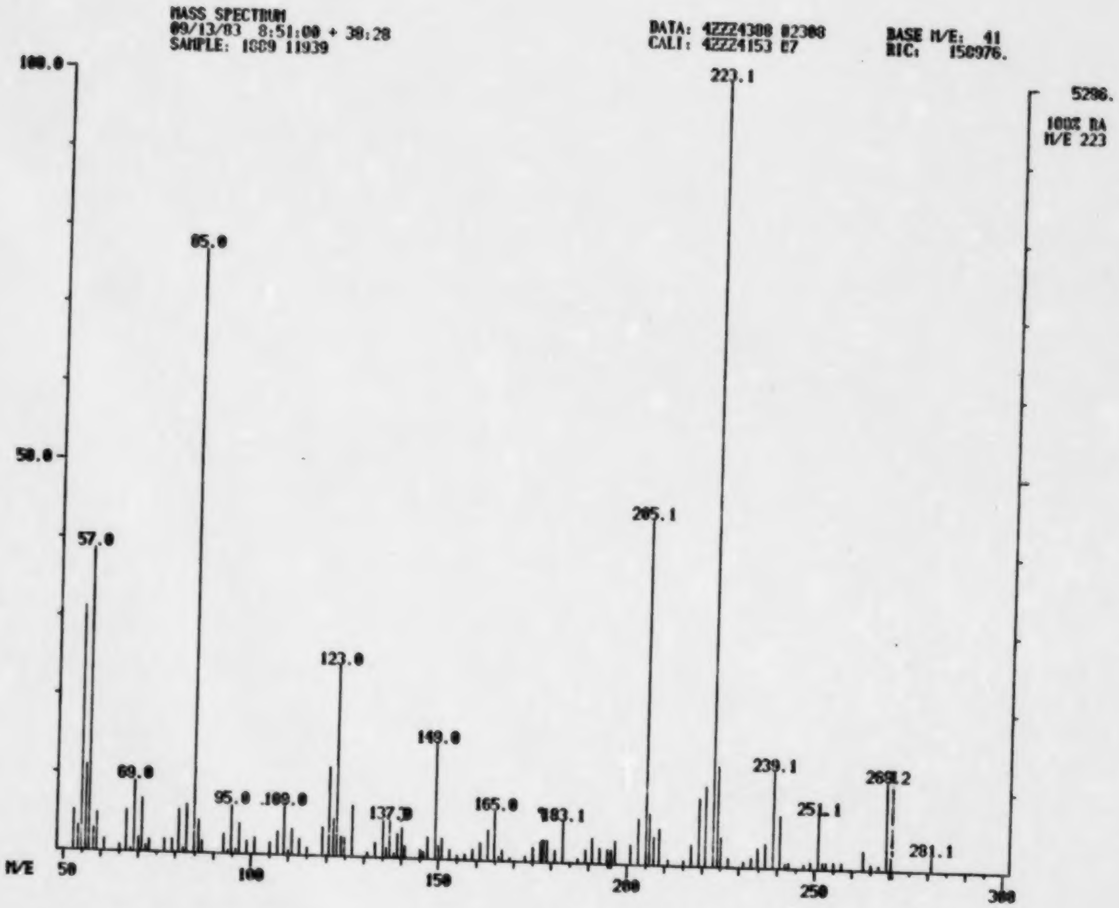


Figure B-131. Chemical ionization mass spectrum, same retention time as EPA compound of interest, scan #2308.

Figure B-132. Conclusions

The ions observed in the electron ionization mass spectrum appear to be fragment ions. Accordingly, chemical ionization mass spectrometry was attempted in order to gain information about a molecular species. The capillary profile for the chemical ionization analysis is shown in Figure B-130, the profile for the EI analysis in Figure B-128. Any correspondence between the two is tenuous, at best. The two chromatograms illustrate the difficulty in extracting meaningful information from chemical ionization analysis of an extremely complex total unknown. Chemical reactions are occurring in the ion source, and there is a competition among the various species present at any given time for the reagent gas. The EPA compound of interest, in this case, is a small chromatographic peak seen above an extensive background. Reactivity of any given compound under chemical ionization conditions is a variable, in any case. It is possible that, if the compound of interest were fractionated or isolated and introduced to chemical ionization mass spectrometry in its pure form, reaction conditions would not be favorable for production of a molecular ion species. In the present case, a comparison of the EI and CI spectra produces no useful information. Without further data than the low resolution mass spectrum, there are no criteria for choosing among the nine possibilities produced by accurate mass measurement for the ion of mass 193. The overall analytical process thus produces information but no conclusions can be reached about composition or structure of the compound of interest.

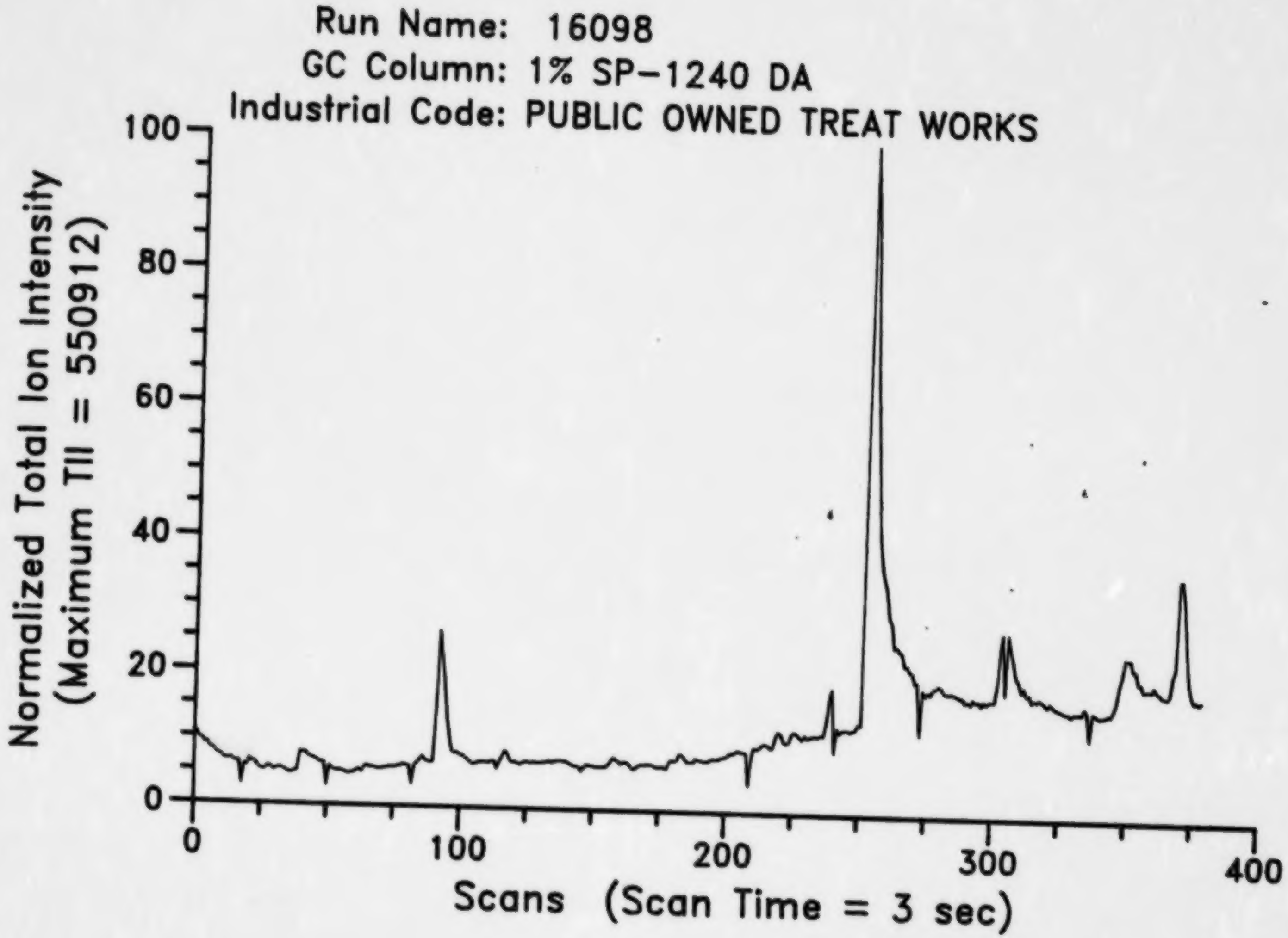


Figure B-133. Packed column chromatogram, extract 16098 (acid), publicly owned treatment works.

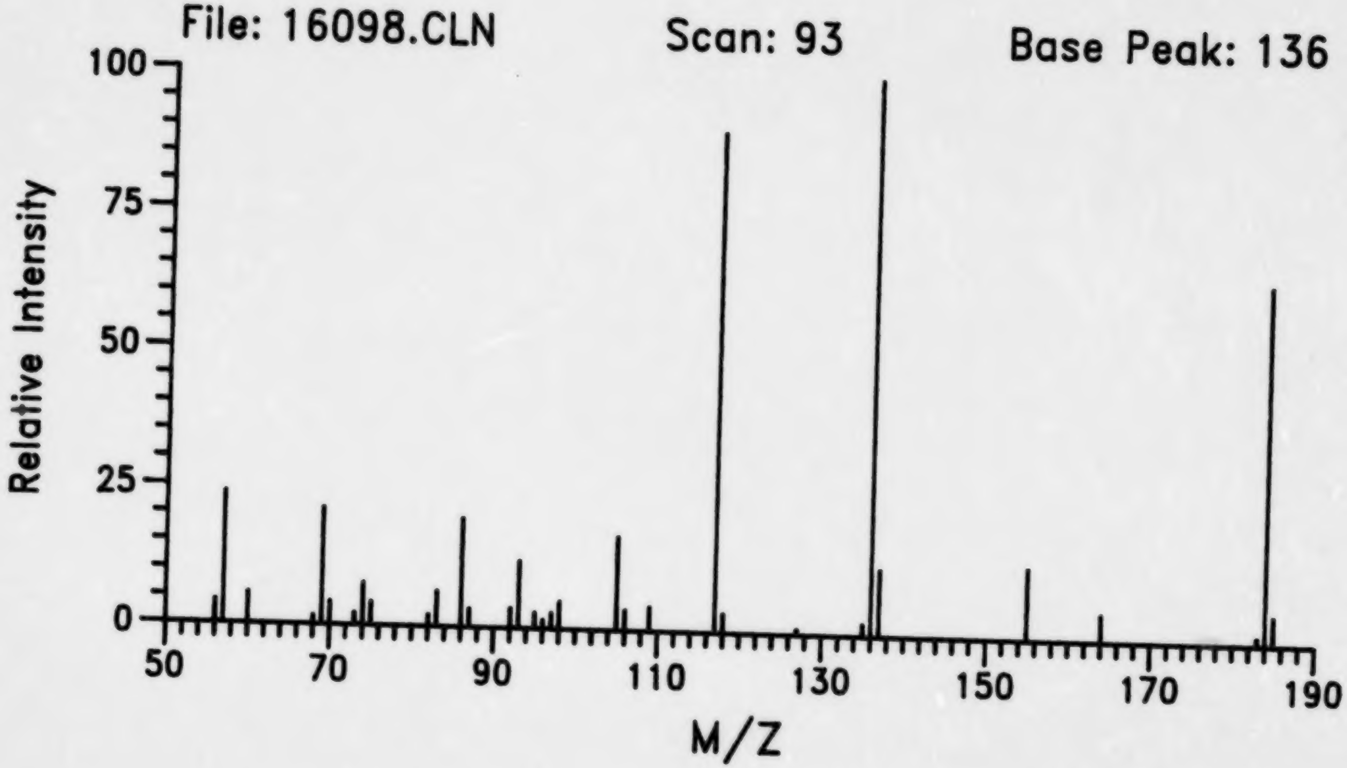


Figure B-134. Extract 16098 (acid), publicly owned treatment works. Frequency of occurrence: 24.

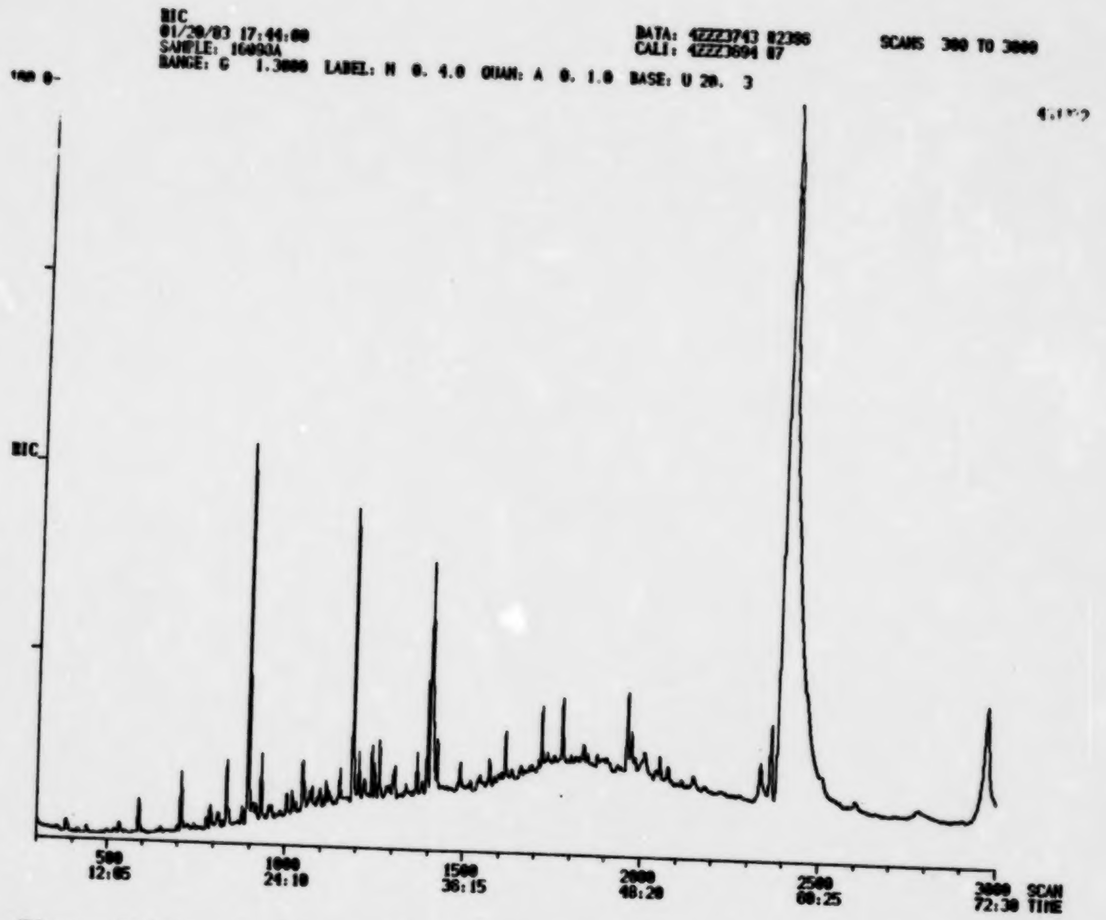


Figure B-135. Capillary chromatogram, extract 16098 (acid), publicly owned treatment works.

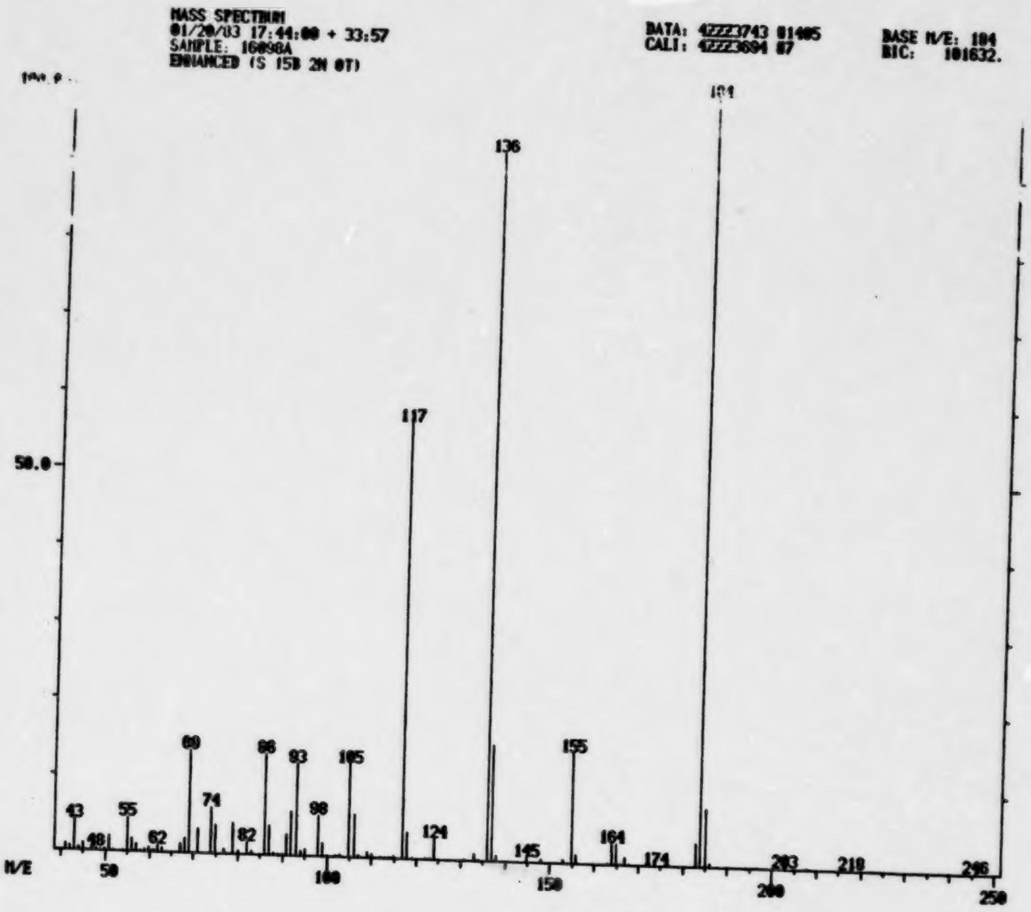


Figure B-136. EPA compound of interest, extract 16098 (acid), publicly owned treatment works.

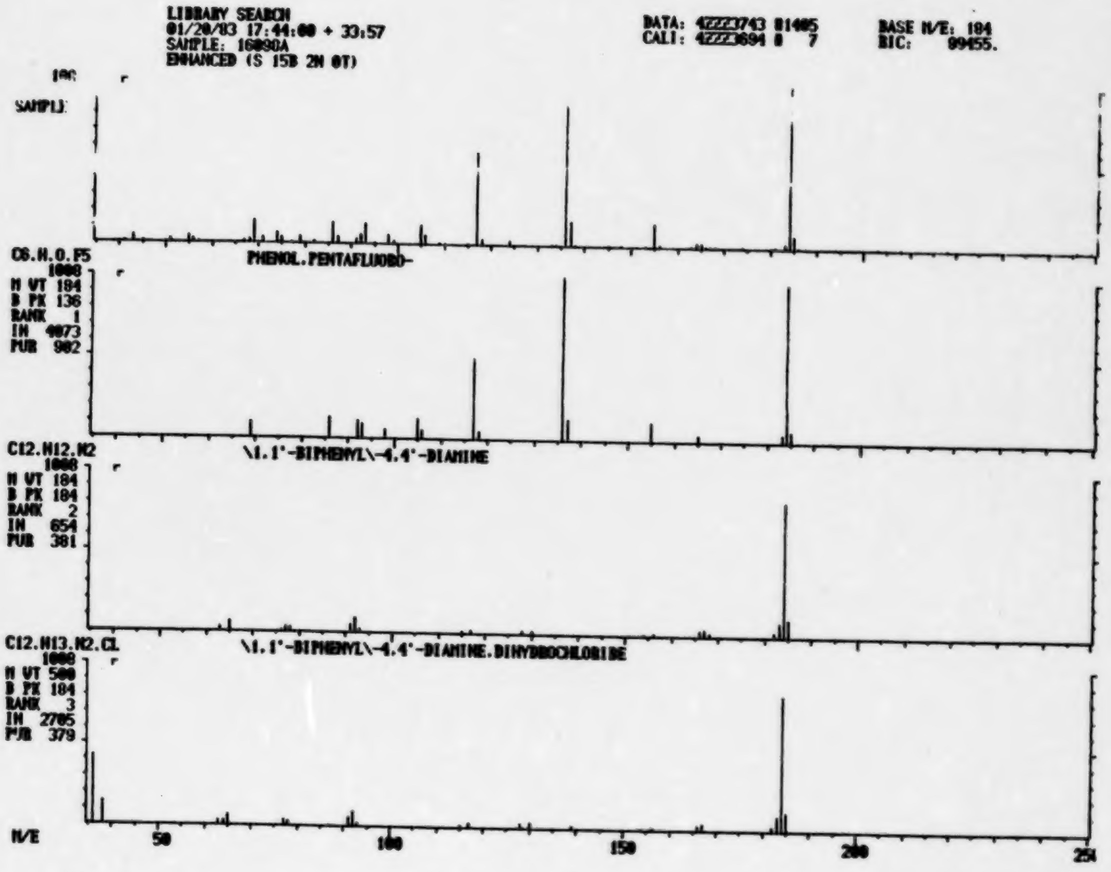


Figure B-137. Computerized library search, EPA compound of interest, extract 16098 (acid), publicly owned treatment works.

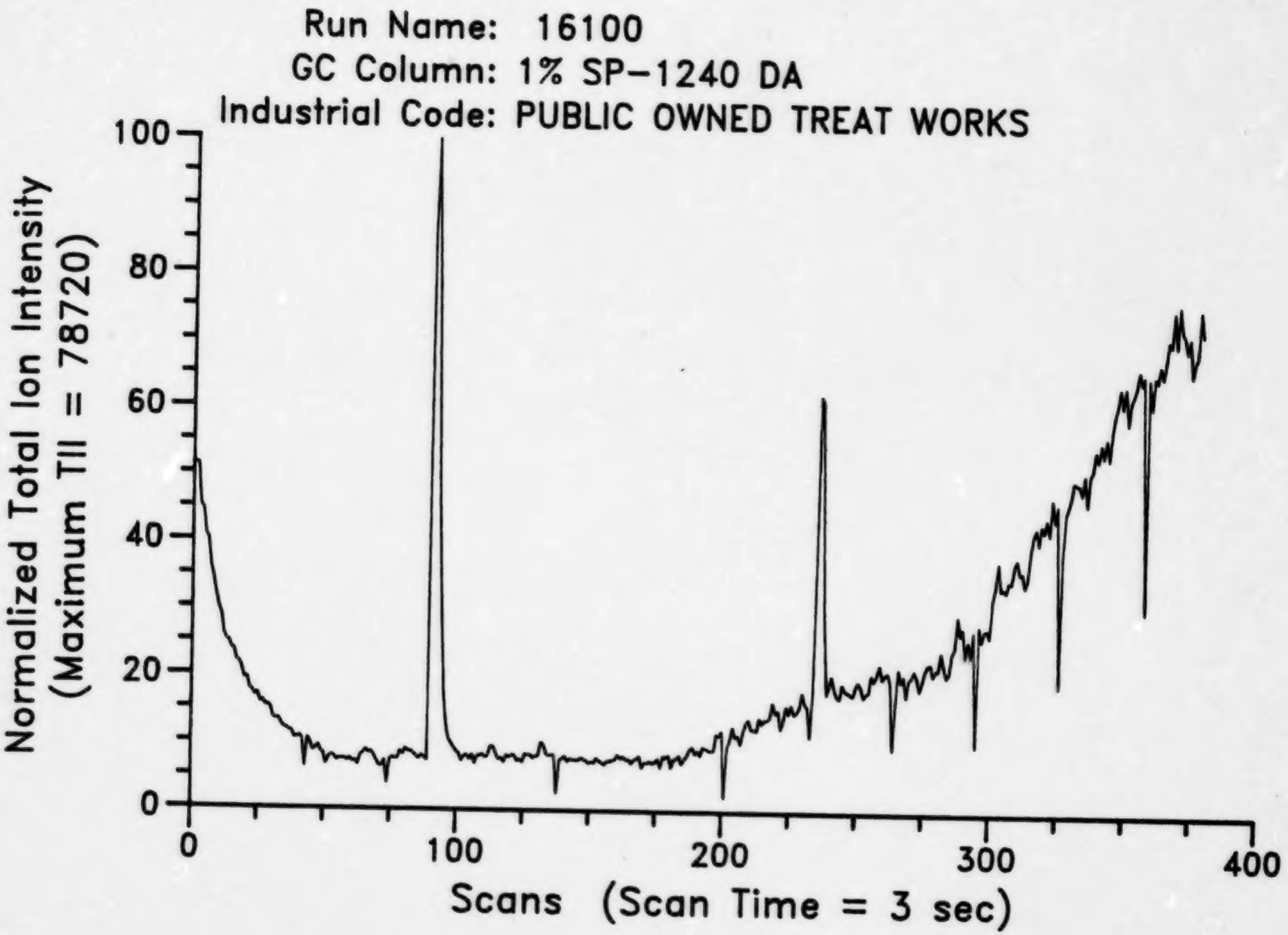


Figure B-138. Packed column chromatogram, extract 16100 (acid), publicly owned treatment works.

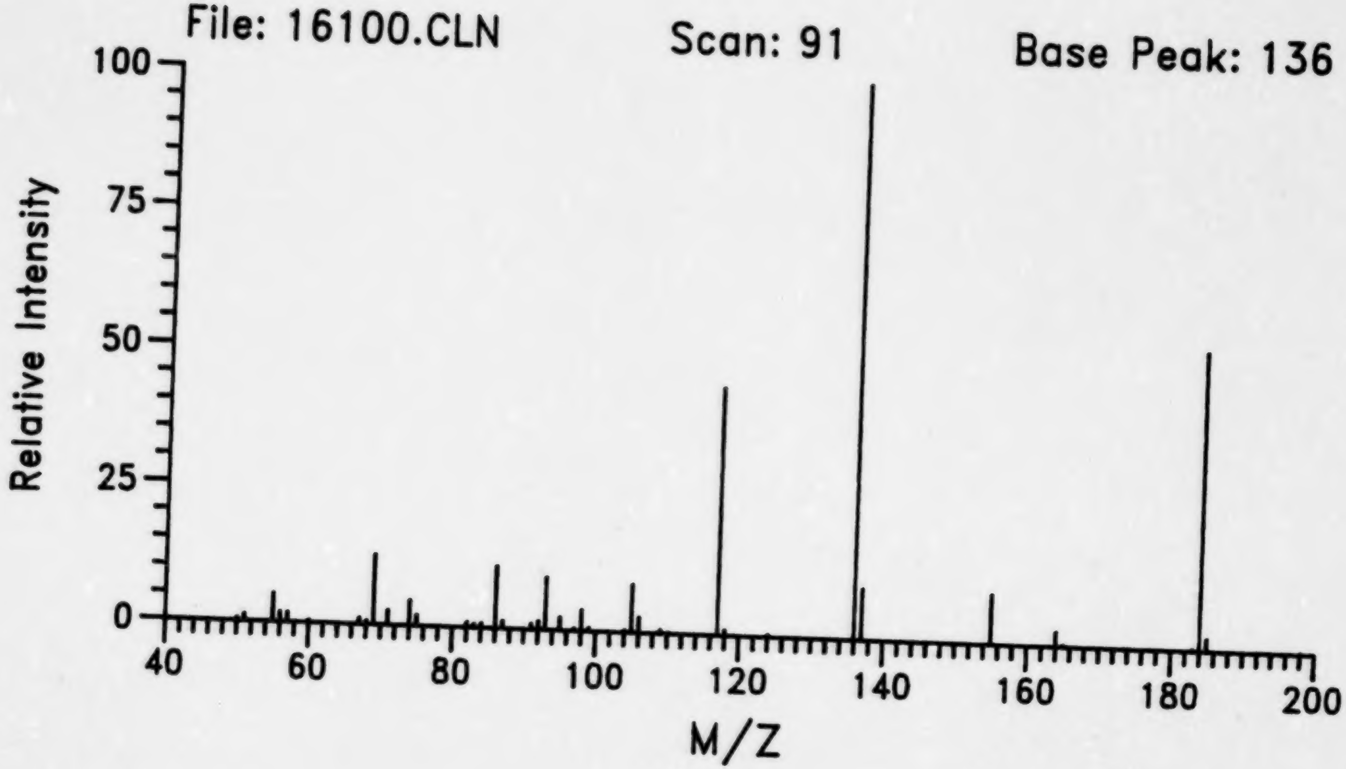


Figure B-139. Extract 16100 (acid), publicly owned treatment works. Frequency of occurrence: 24.

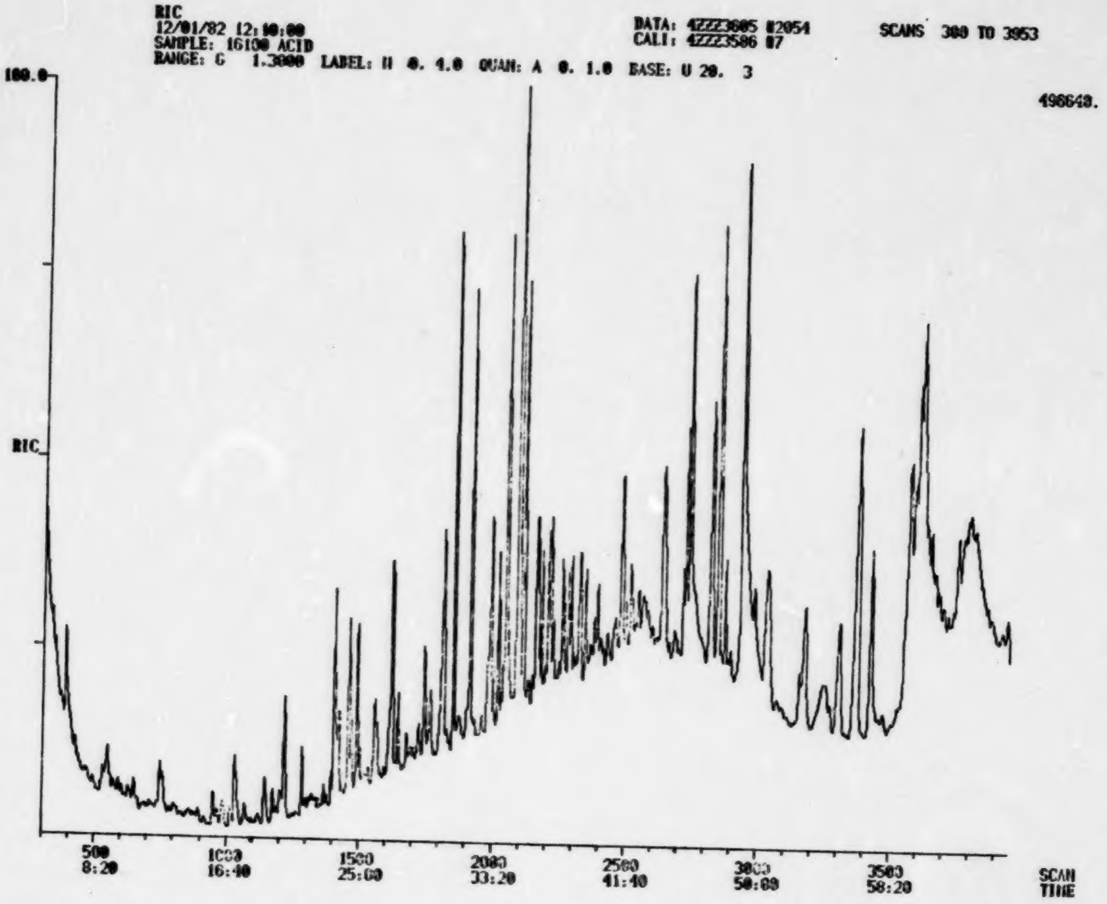


Figure B-140. Capillary chromatogram, extract 16100 (acid), publicly owned treatment works.

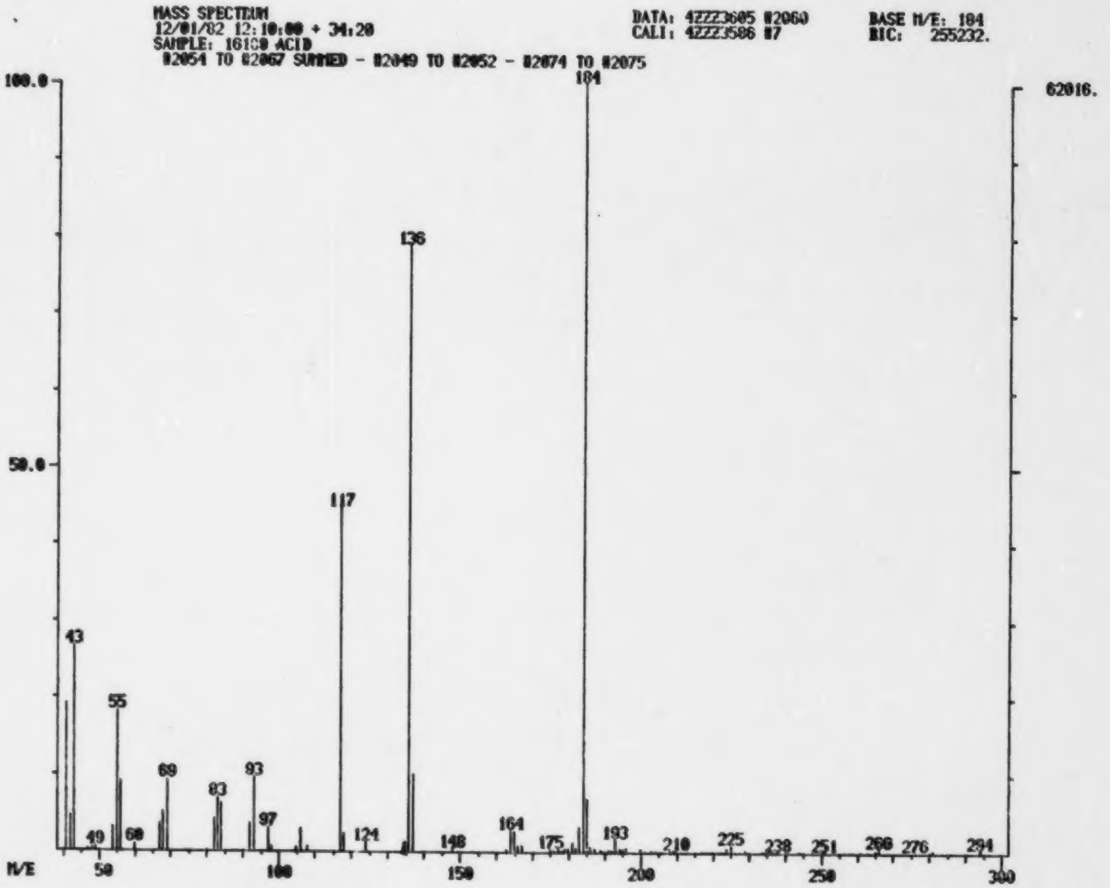


Figure B-141. Mass spectrum of EPA compound of interest, extract 16100 (acid), publicly owned treatment works, scan #2060.

Figure B-142. Conclusions

The EPA compound of interest is obviously the same compound in the two extracts: the mass spectra are the same and the capillary retention times correspond within 20 seconds. Accurate mass measurement presents two possibilities: C_6HOF_5 (error, -1.0 mmu) and $C_3H_2O_2F_6$ (error, 0.2 mmu). A computerized library search of the mass spectrum obtained from the capillary assay identifies the compound as pentaflurophenol, with FIT of 994 (see Figure B-137). Identification as pentafluorophenol is consistent with the data obtained from accurate mass measurement. It would also be reasonable for pentafluorophenol to be found in the acid fraction of the extract. The other possibility from accurate mass measurement, $C_3H_2O_2F_6$, would be consistent with a postulated structure as a hexafluorinated diol. This postulation as a diol raises questions about the stability of the compound in water. The more probable identification for the compound is pentaflurophenol.

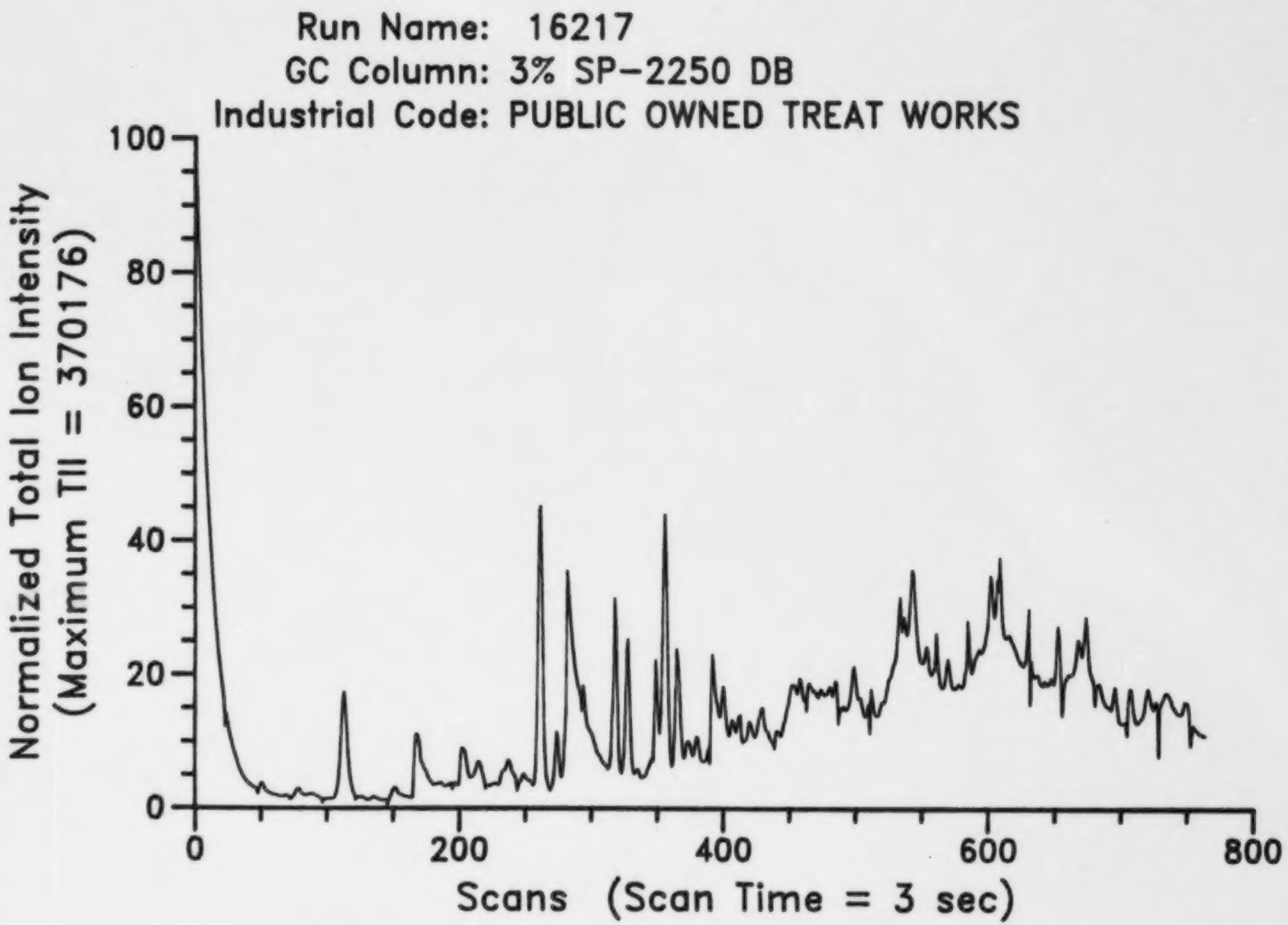


Figure B-143. Packed column chromatogram, extract 16217 (base/neutral), publicly owned treatment works.

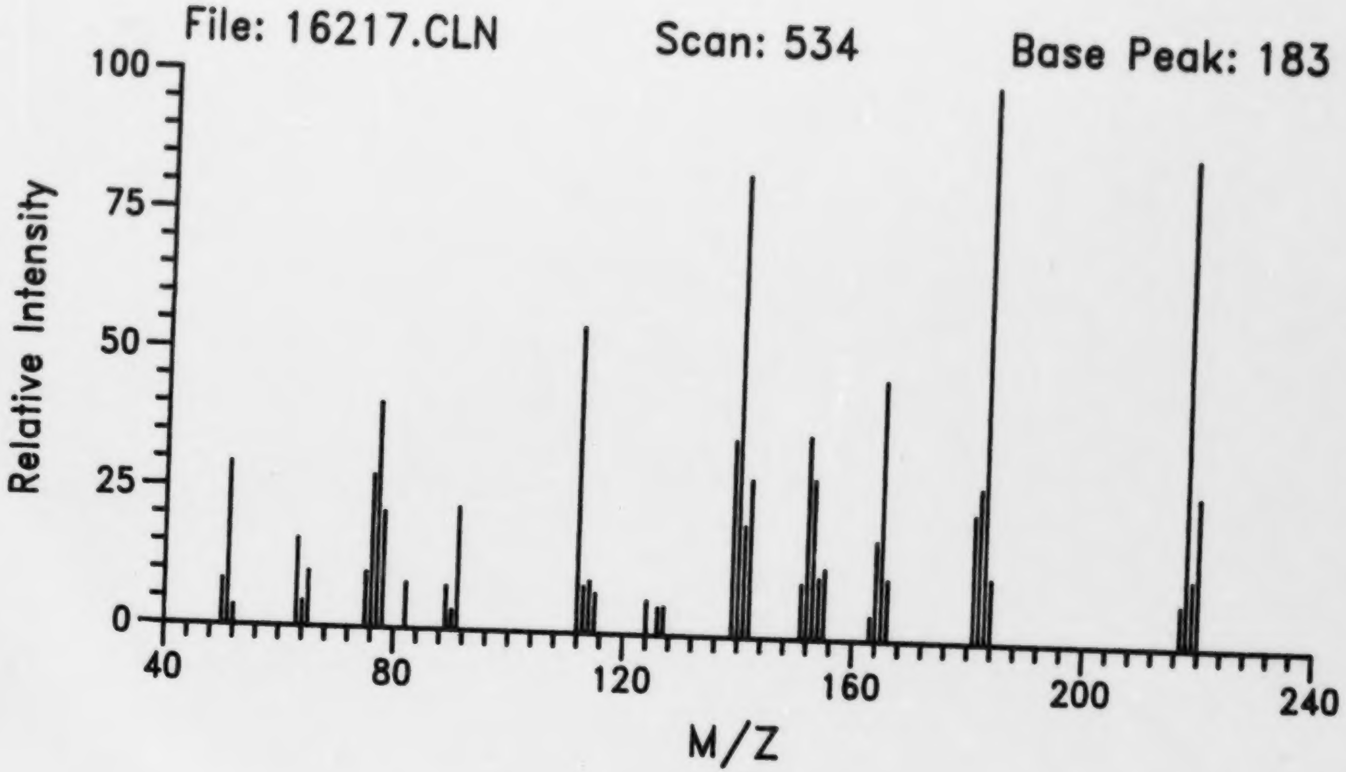


Figure B-144. Extract 16217 (base/neutral), publicly owned treatment works. Frequency of occurrence: 11.

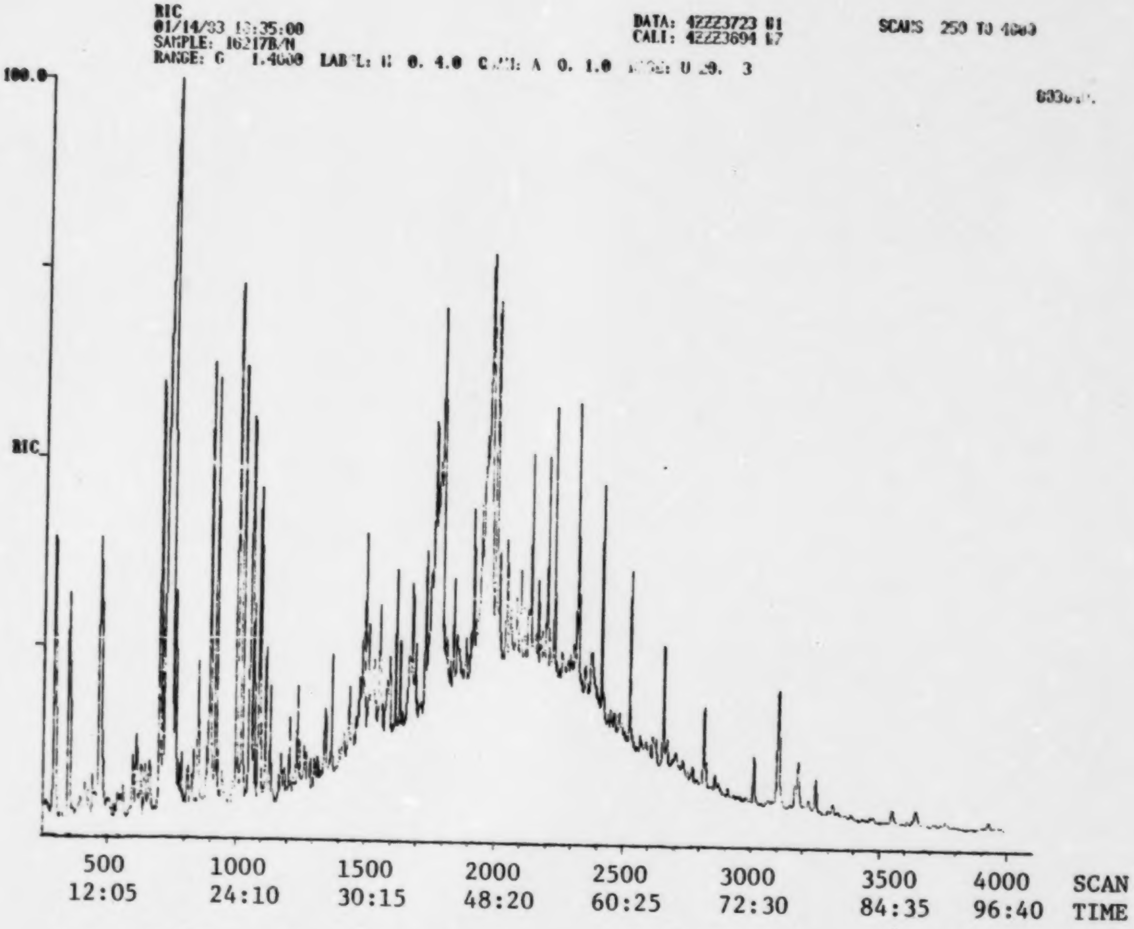


Figure B-145. Capillary chromatogram, extract 16217 (base/neutral), publicly owned treatment works.

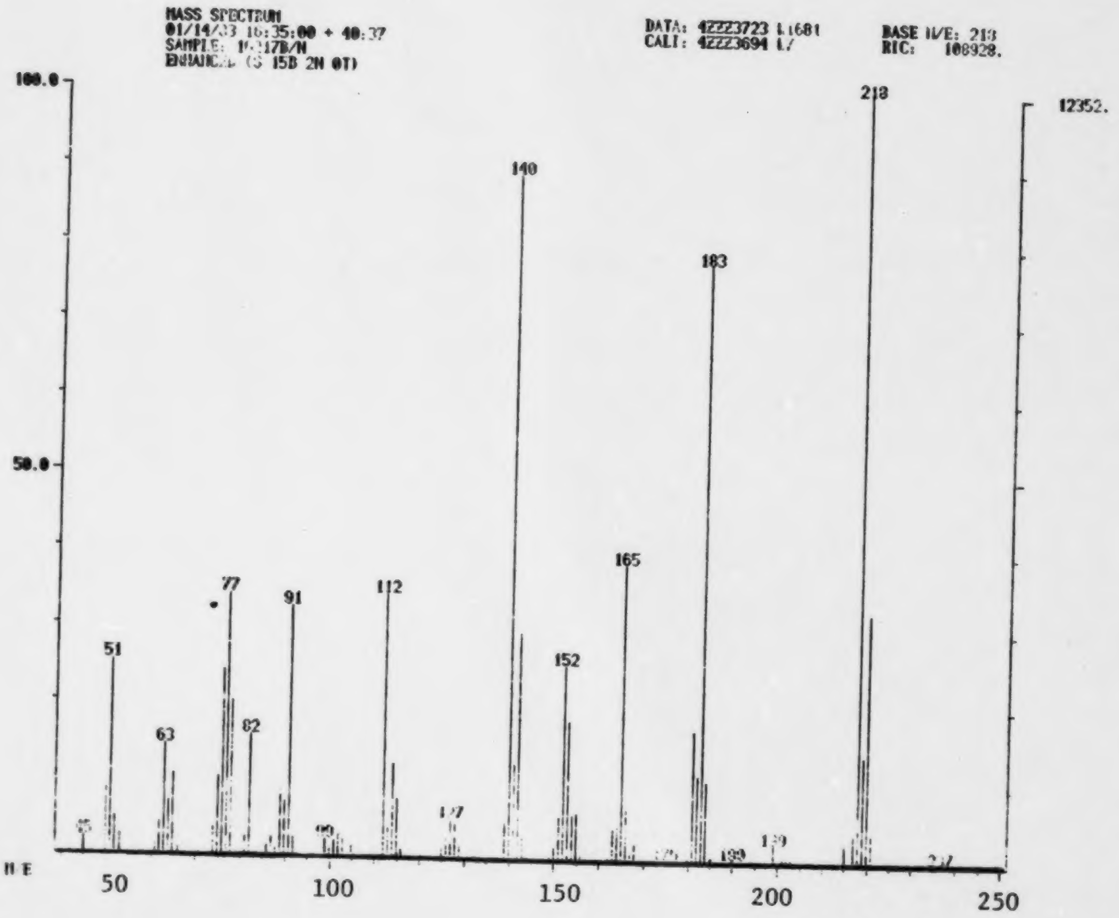


Figure B-146. Mass spectrum, EPA compound of interest, extract 16217 (base/neutral), publicly owned treatment works, scan #1681.

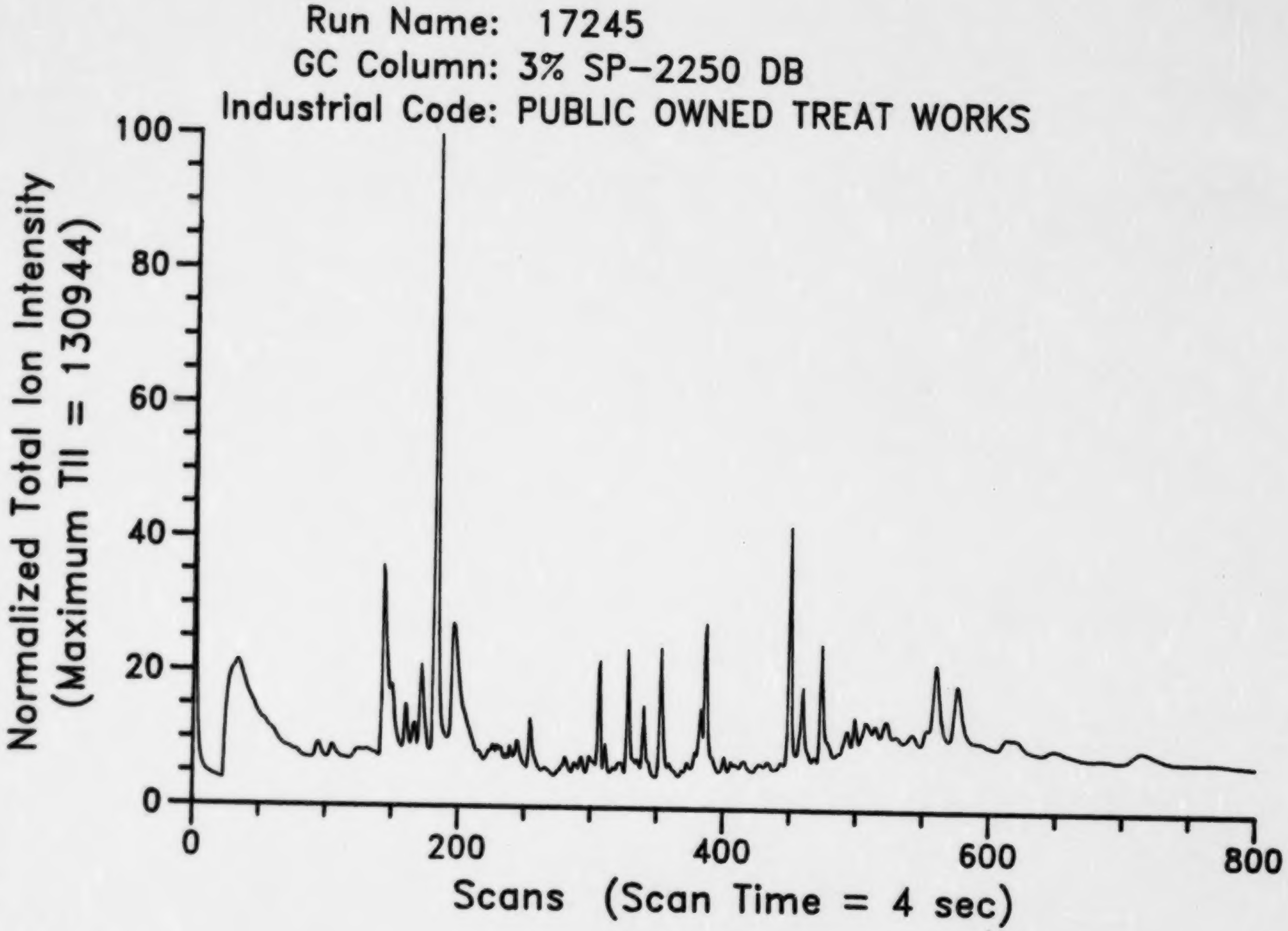


Figure B-147. Packed column chromatogram, extract 17245 (base/neutral), publicly owned treatment works.

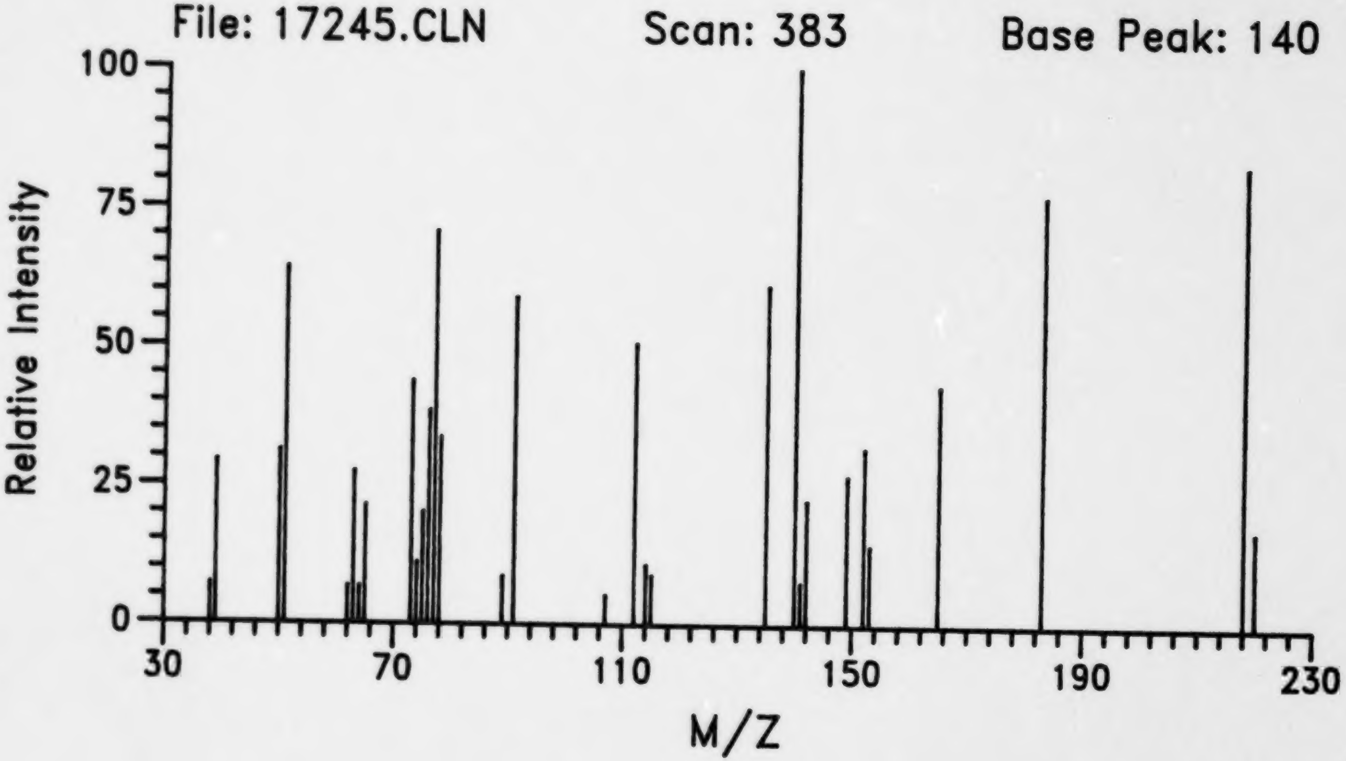


Figure B-148. Extract 17245 (base/neutral), publicly owned treatment works. Frequency of occurrence: 11.

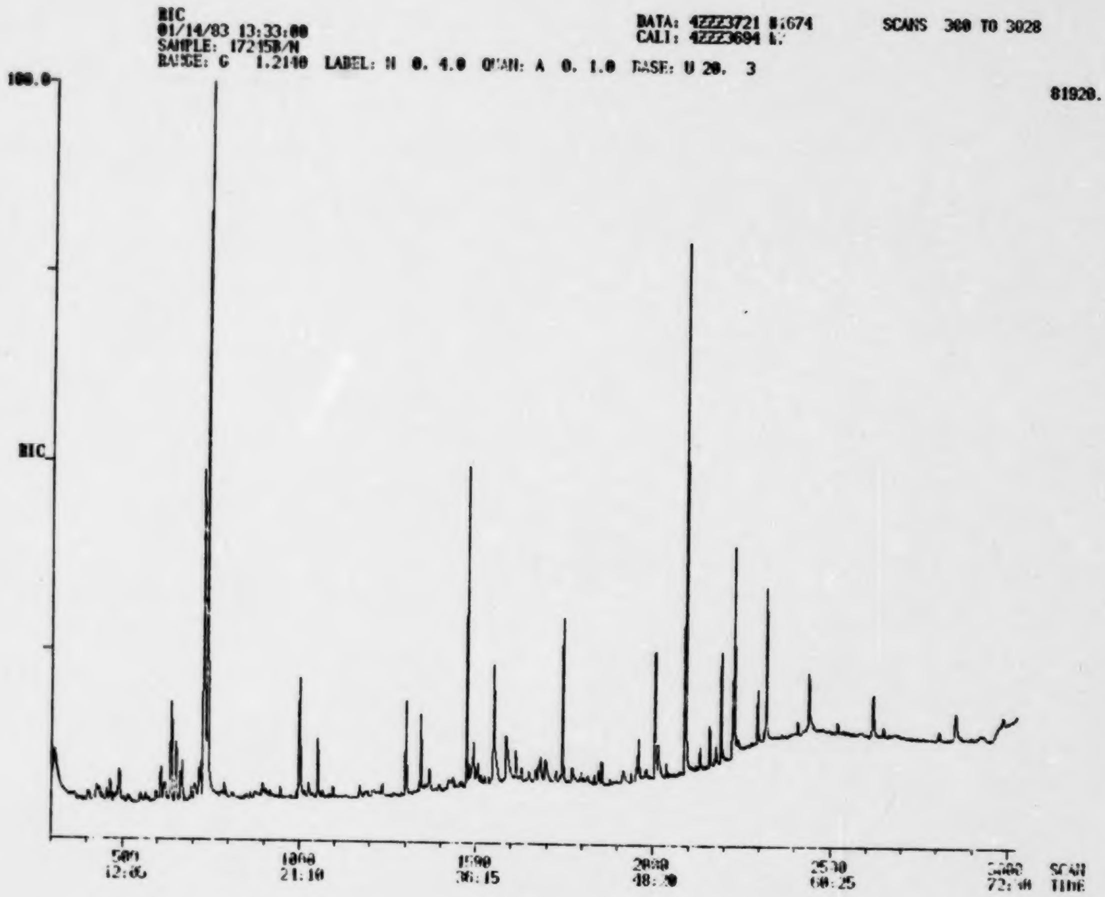


Figure B-149. Capillary chromatogram, extract 17245 (base/neutral), publicly owned treatment works.

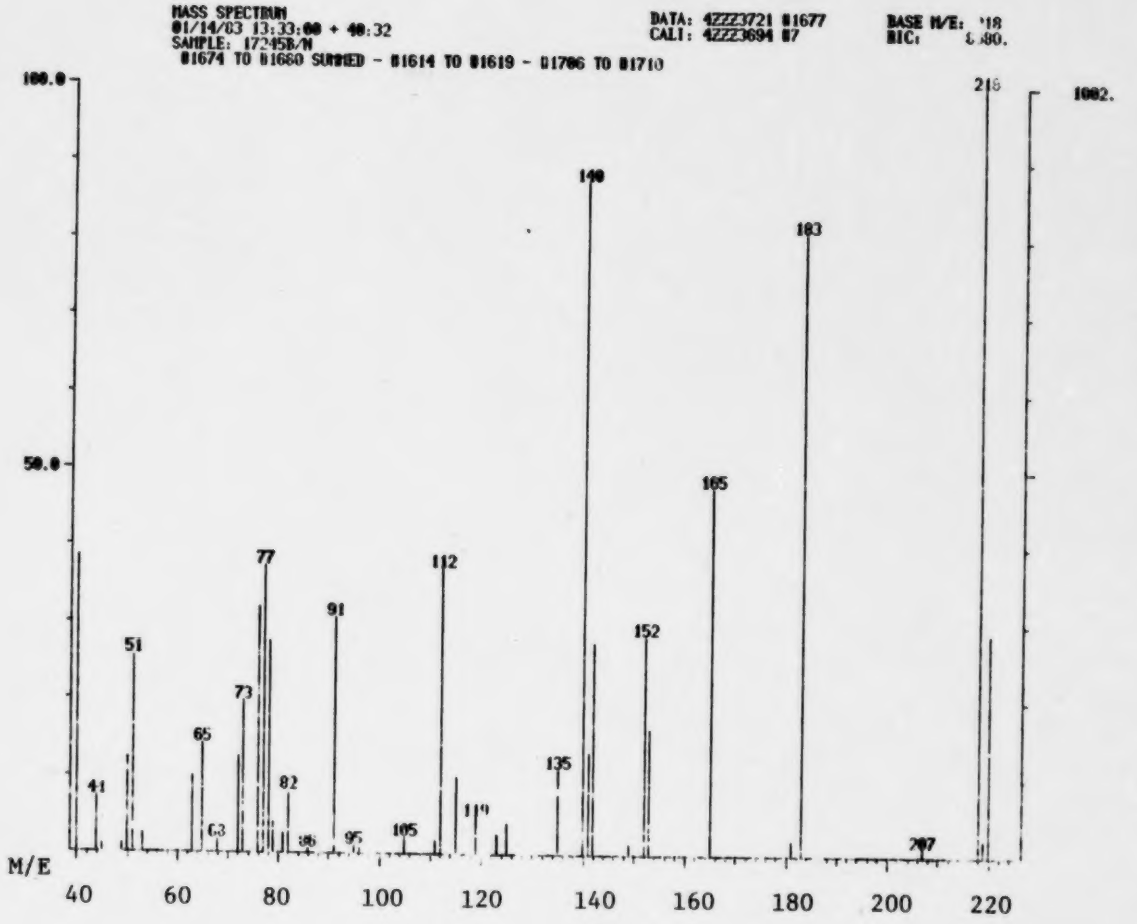


Figure B-150. Mass spectrum, EPA compound of interest, extract 16245 (base/neutral), publicly owned treatment works, scan #1677.

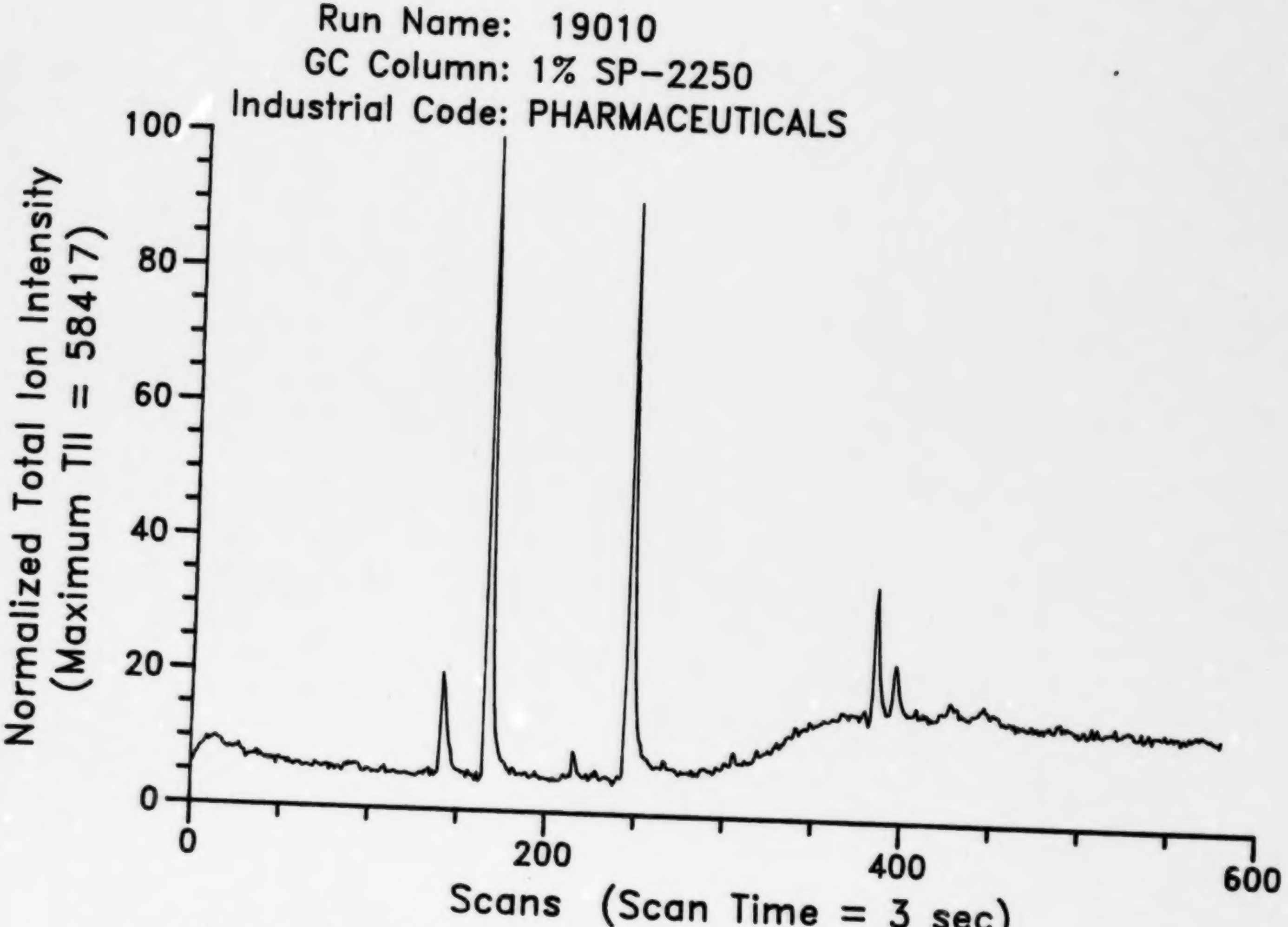


Figure B-151. Packed column chromatogram, extract 19010 (base/neutral), pharmaceuticals industry.

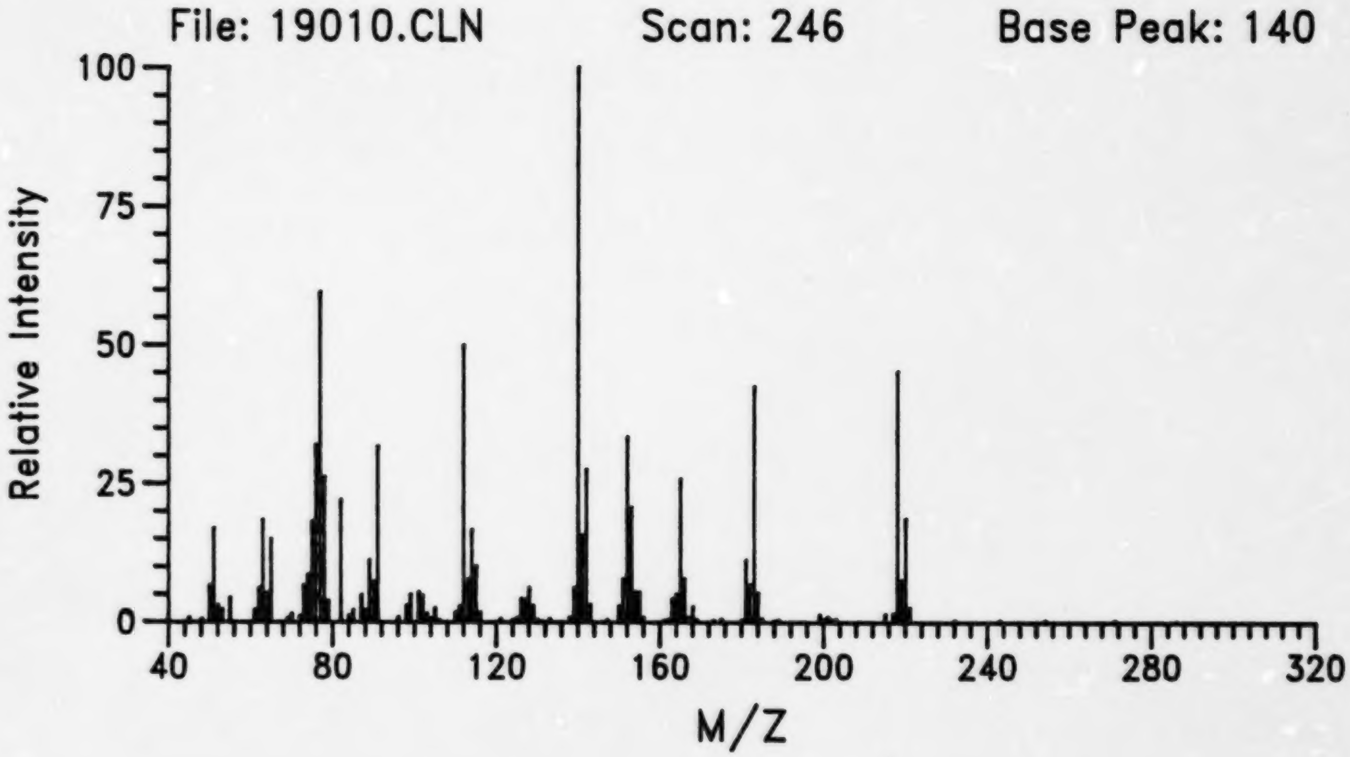


Figure B-152. Extract 19010 (base/neutral), pharmaceuticals industry.
Frequency of occurrence: 11.

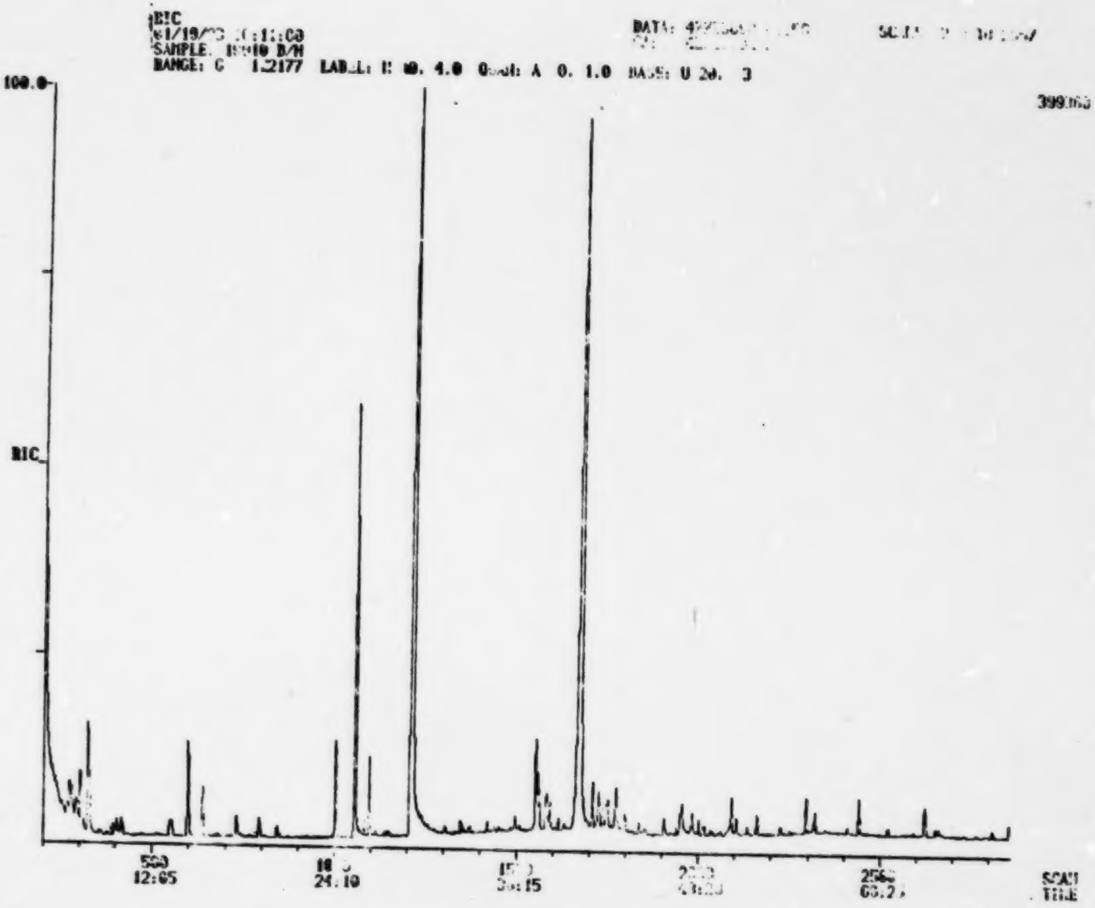


Figure B-153. Capillary chromatogram, extract 19010 (base/neutral), pharmaceuticals industry.

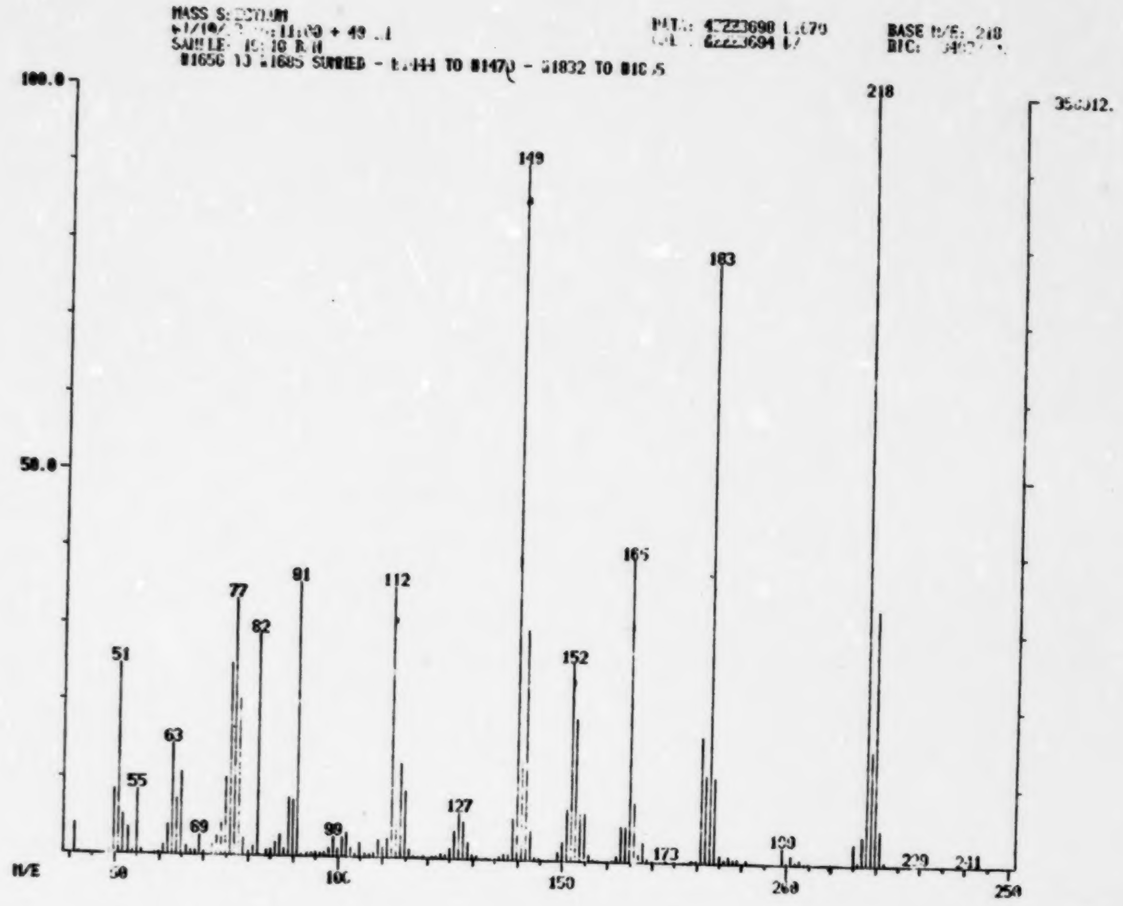
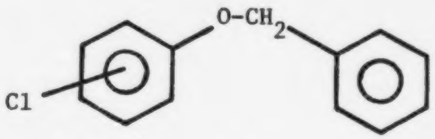


Figure B-154. Mass spectrum, EPA compound of interest, extract 19010 (base/neutral), pharmaceuticals industry, scan #1679.

Figure B-155. Conclusions

The molecular ion at mass 218 appears to contain one chlorine atom, an observation borne out by data from accurate mass measurement which identify the ion as $C_{13}H_{11}OCl$ (error, 5.0 mmu) and the ion at mass 183, which does not contain the chlorine atom any longer, as $C_{13}H_{11}O$ (error, -4.6 mmu). The ions at lower mass in the spectrum support the postulation of a benzene ring as part of the structure of the molecule. The ions at mass 112 and 114 support the postulation that a chlorine atom is attached to the benzene ring. A structure such as the one below is consistent with the mass assignments and mass spectrometric peaks, but no standards are available.



Analysis by FT-IR or NMR would be able to prove or disprove this postulation, but sample quantity is not sufficient.

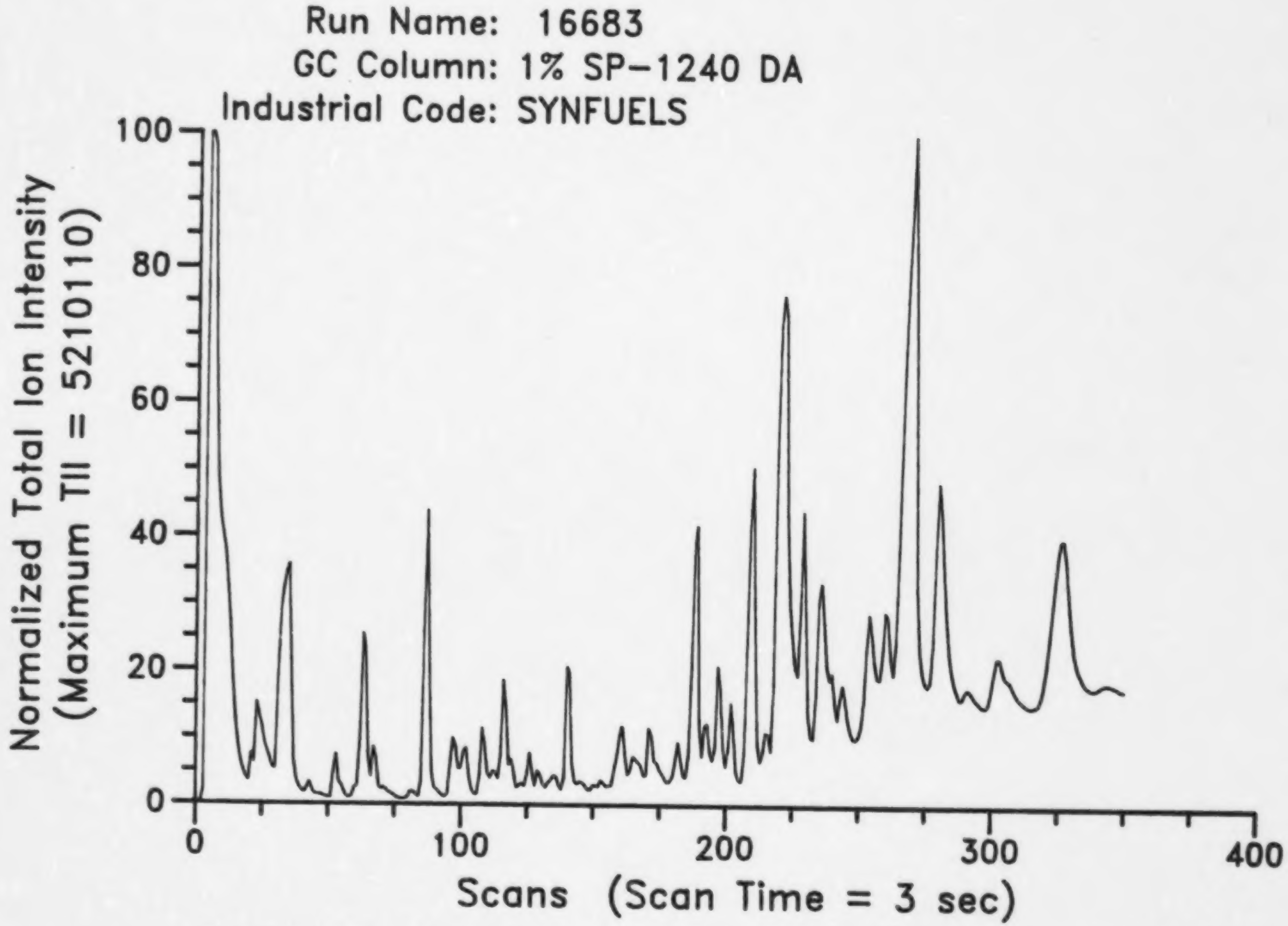


Figure B-156. Packed column chromatogram, extract 16683 (acid), synfuels industry.

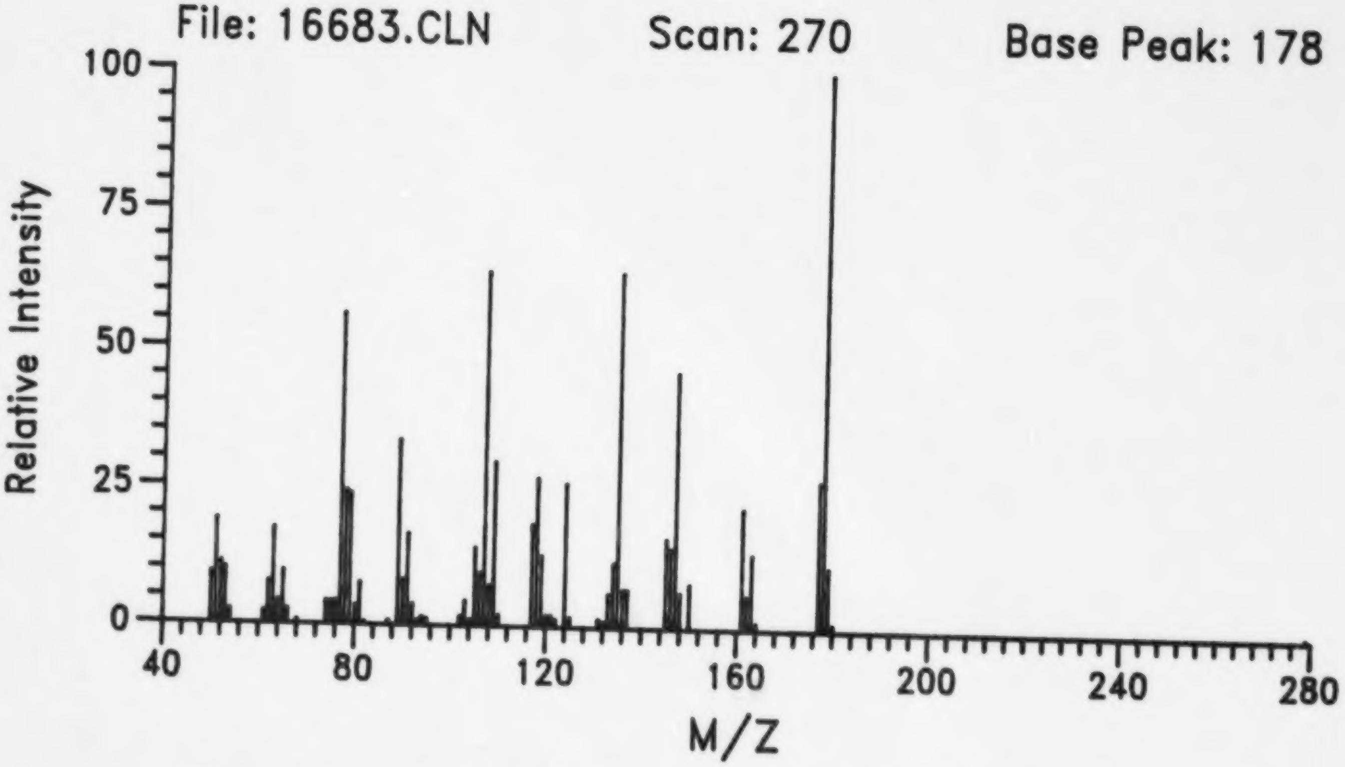


Figure B-157. Extract 16683 (acid), synfuels industry. Frequency of occurrence: 12.

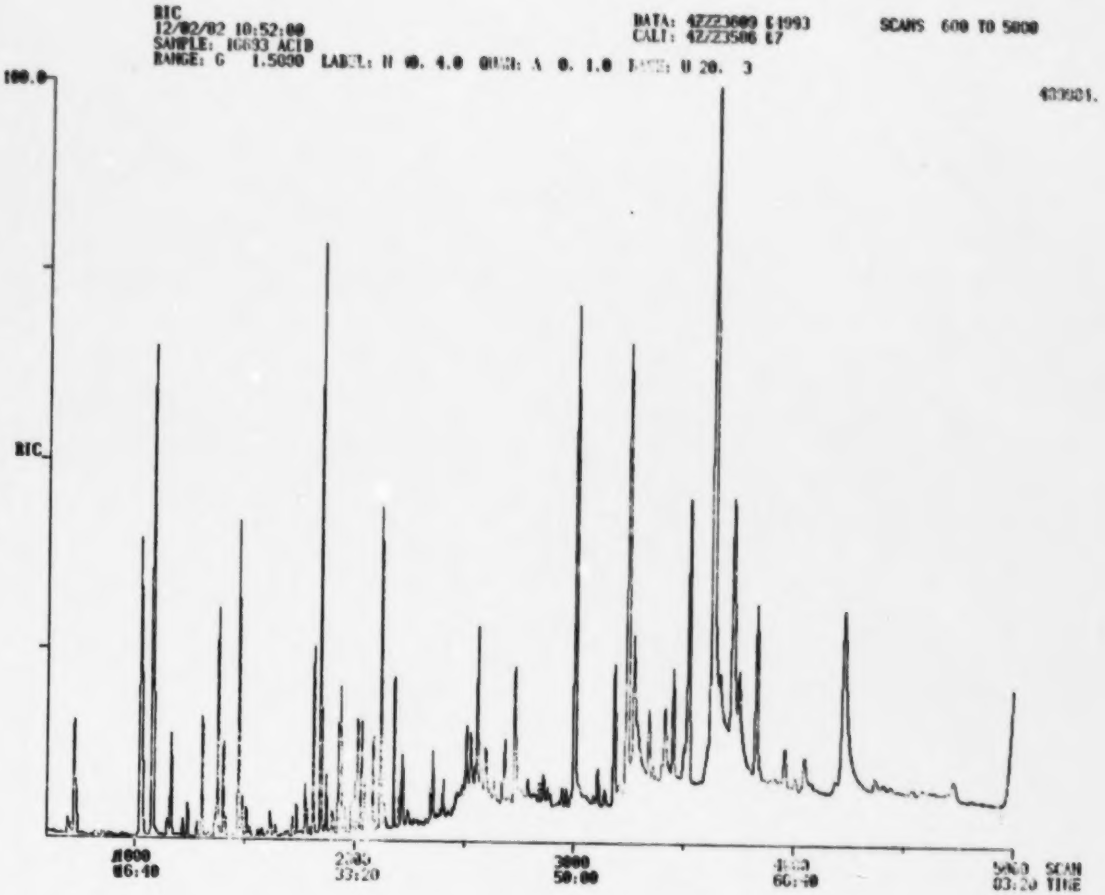


Figure B-158. Capillary chromatogram, extract 16683 (acid), synfuels industry.

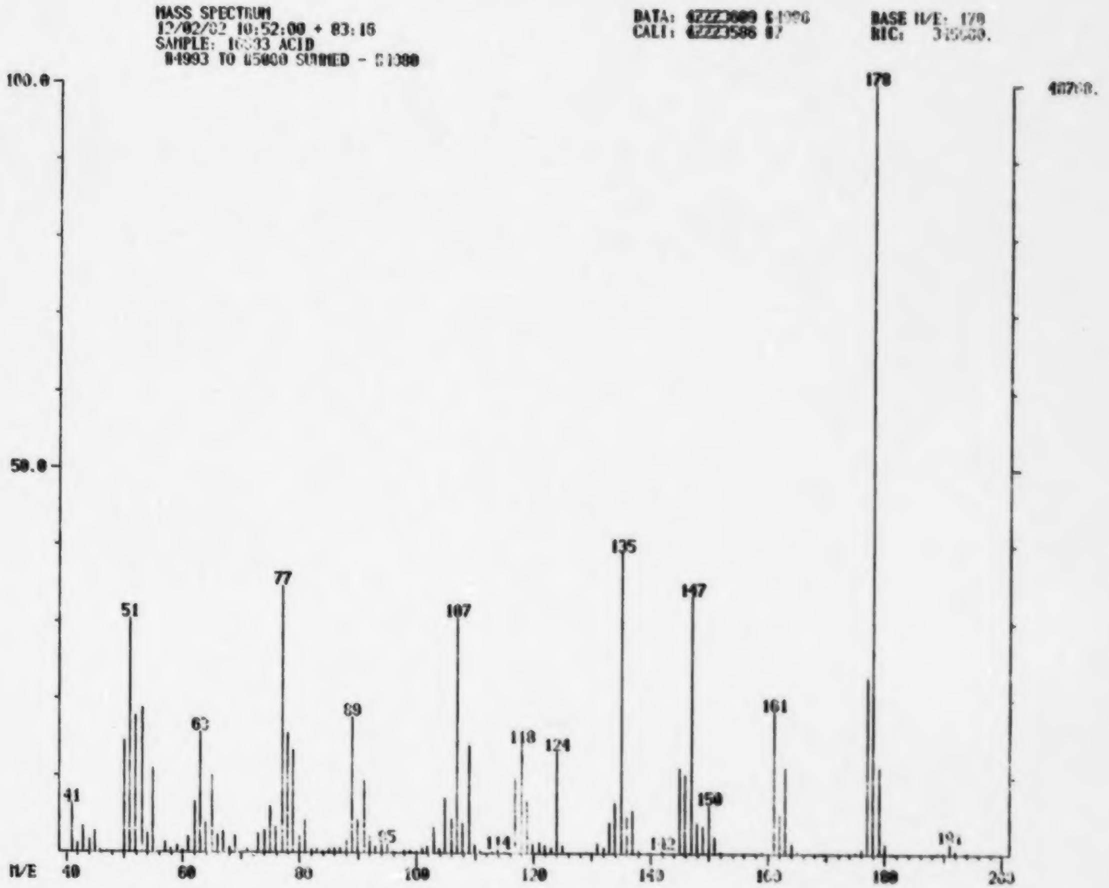


Figure B-159. Mass spectrum, EPA compound of interest, extract 16683 (acid), synfuels industry, scan #4996.

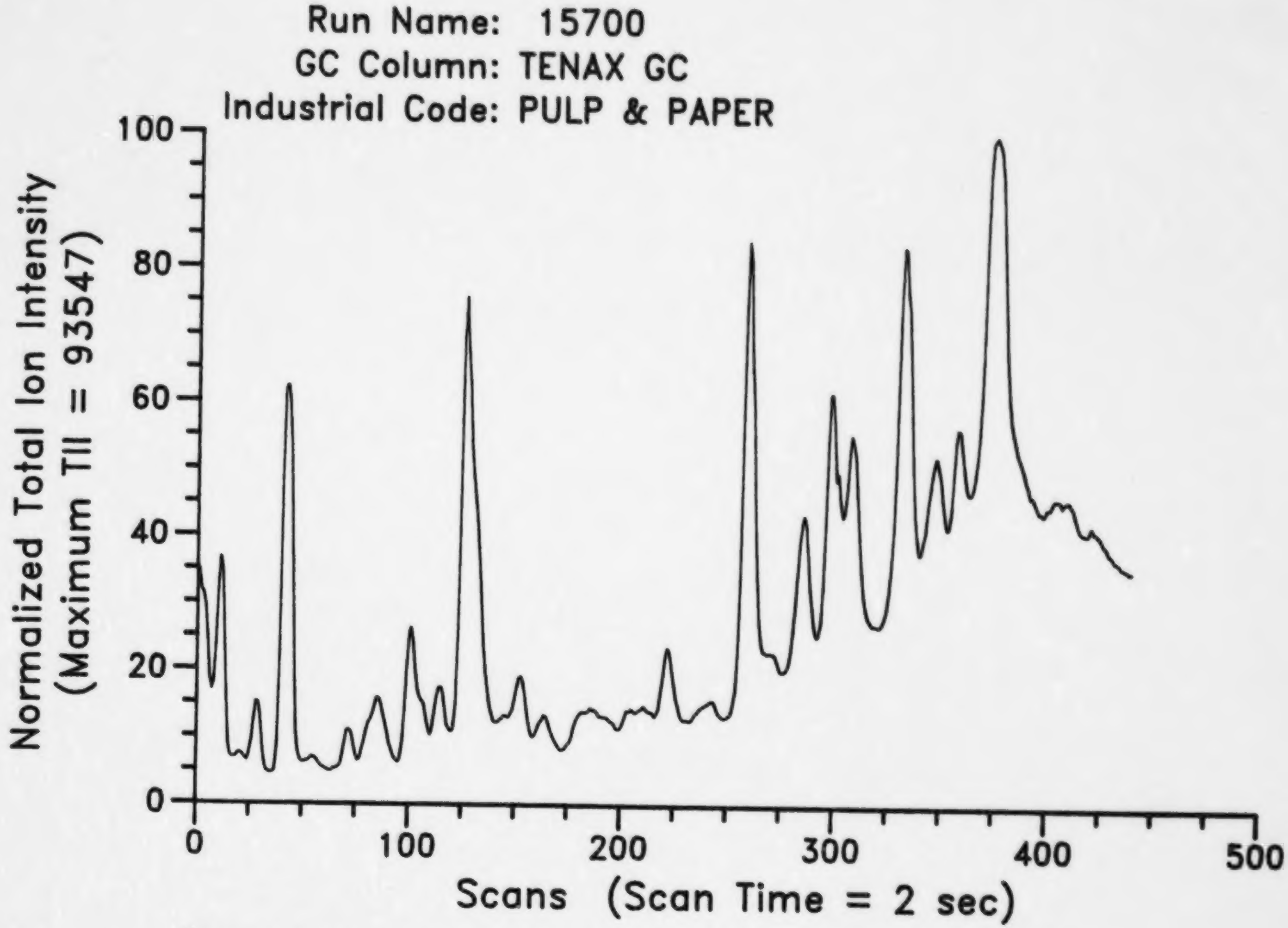


Figure B-160. Packed column chromatogram, extract 15700 (acid), pulp and paper industry.

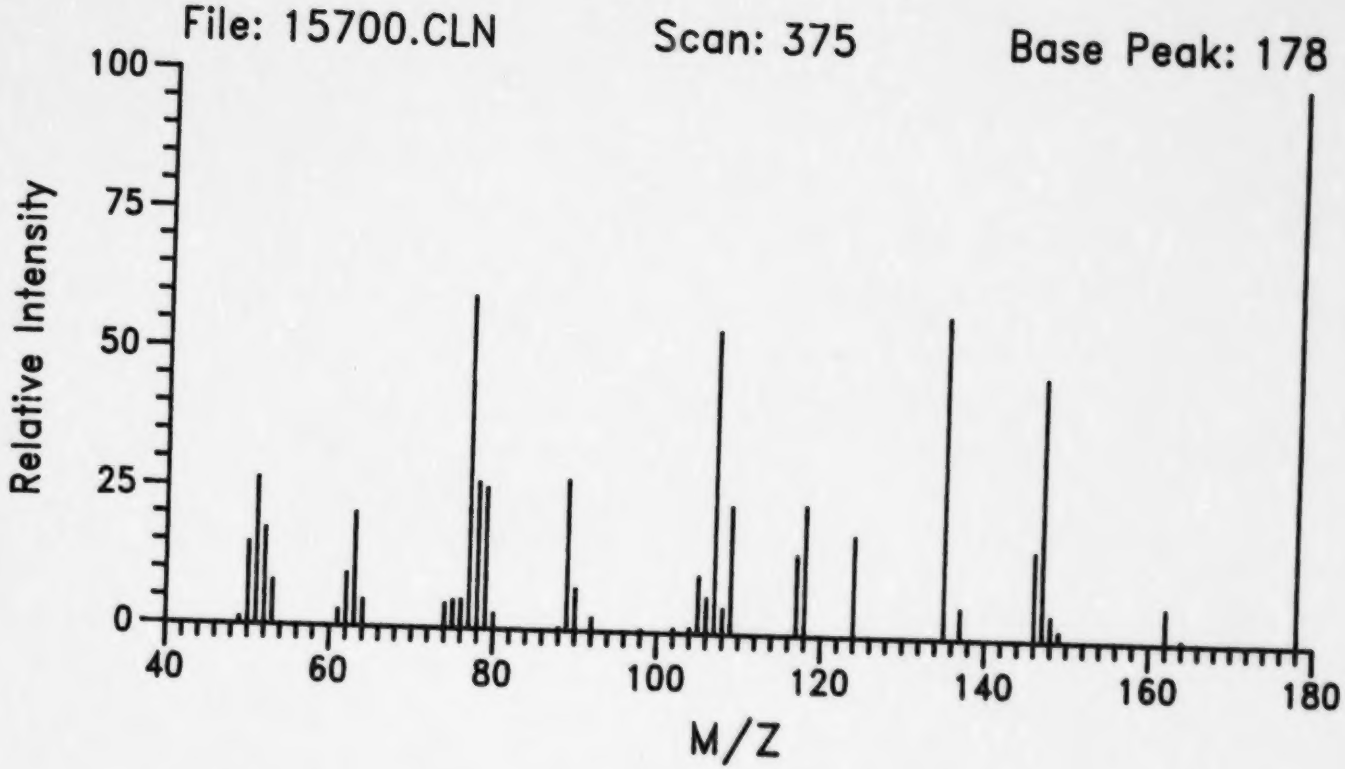


Figure B-161. Extract 15700 (acid), pulp and paper industry. Frequency of occurrence: 12.

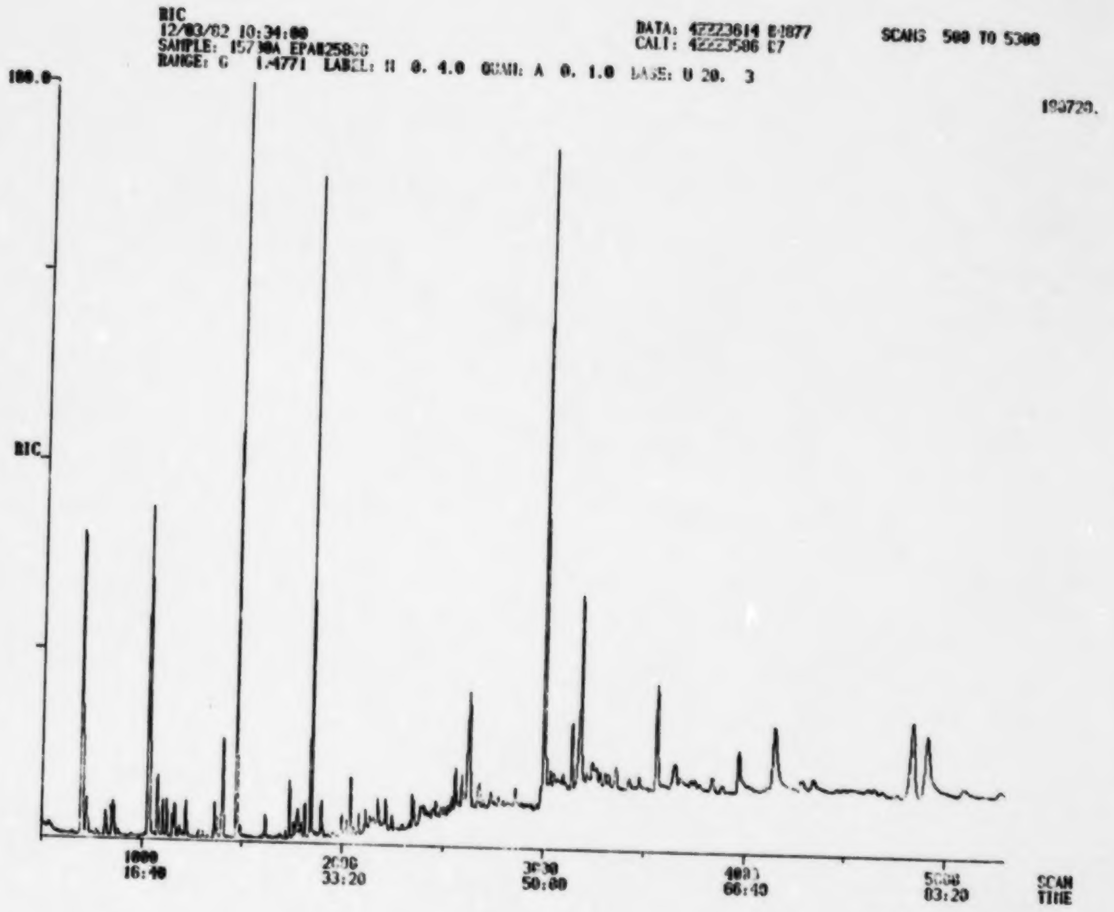


Figure B-162. Capillary chromatogram, extract 15700 (acid), pulp and paper industry.

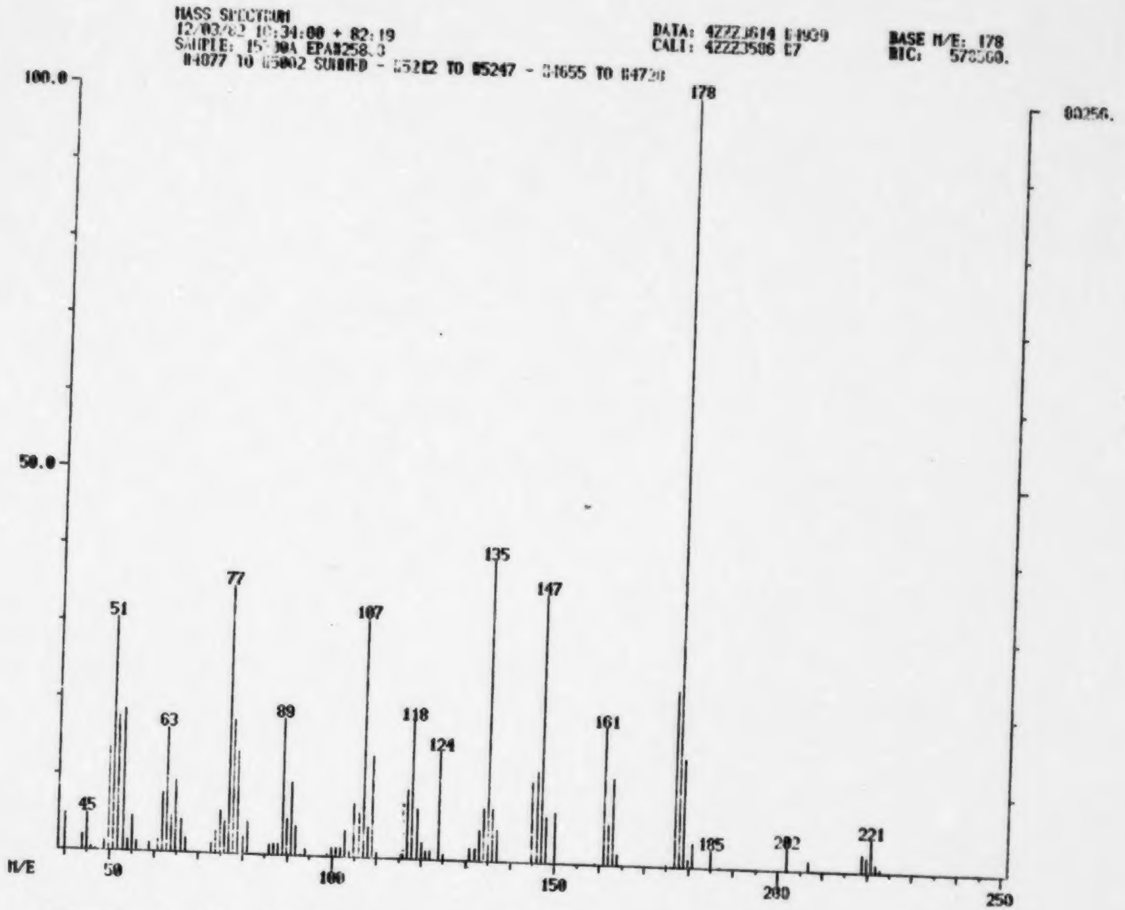
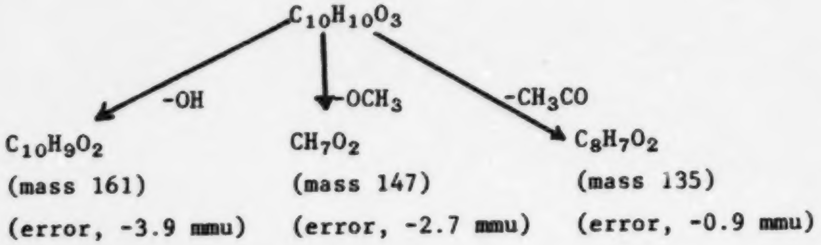


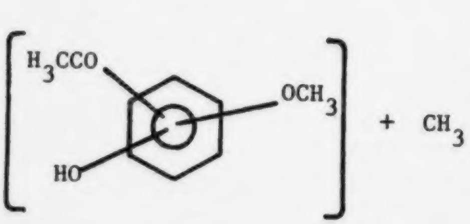
Figure B-163. Mass spectrum, EPA compound of interest, extract 15700 (acid), pulp and paper industry, scan #4939.

Figure B-164. Conclusions

Accurate mass measurement data, in conjunction with some interpretation of the mass spectrum, allow a tentative assignment of structure for the compound of interest. The low mass ions of the mass spectrum indicate the presence of a benzene ring, so all accurate mass measurement possibilities which do not contain at least six carbon atms must be elinated. Two possibilities remain. If $C_9H_{10}N_2O_2$ is viable , to produce a fragment of mass 135 a loss of N_2 and $-CH_3$must occur. Loss of N_2 is not unheard of in organic mass spectrometry, but it is a rare and unusual process and usually involves molecules with a bond between nitrogen atoms, often a multiple bond between these atoms. The likelihood of such a molecule being stable in water and surviving the extraction and storage process is slight. The surviving possibility from accurate mass measurement is $C_{10}H_{10}O_3$ (error, -1.6 mmu) with the following scheme operative in producing the fragments encountered in the mass spectrum.


$C_{10}H_{10}O_3$
-OH
$-OCH_3$
$-CH_3CO$
$C_{10}H_9O_2$
(mass 161)
(error, -3.9 mmu)
CH_7O_2
(mass 147)
(error, -2.7 mmu)
$C_8H_7O_2$
(mass 135)
(error, -0.9 mmu)

The above considerations make postulations of the following partial structure a logical possibility.


H_3CCO
OCH_3
HO
+ CH_3

Several elements of doubt remain. It is possible that the methyl group might be attached directly to the benzene ring, but in this case, a more intense peak at $\underline{m}/\underline{z}$ 91 might be anticipated. Evidence from FT-IR about the exact nature of the functional groups and their positions of attachment to the aromatic ring would be extremely valuable, but this information is not available because of the small amount of the compound.

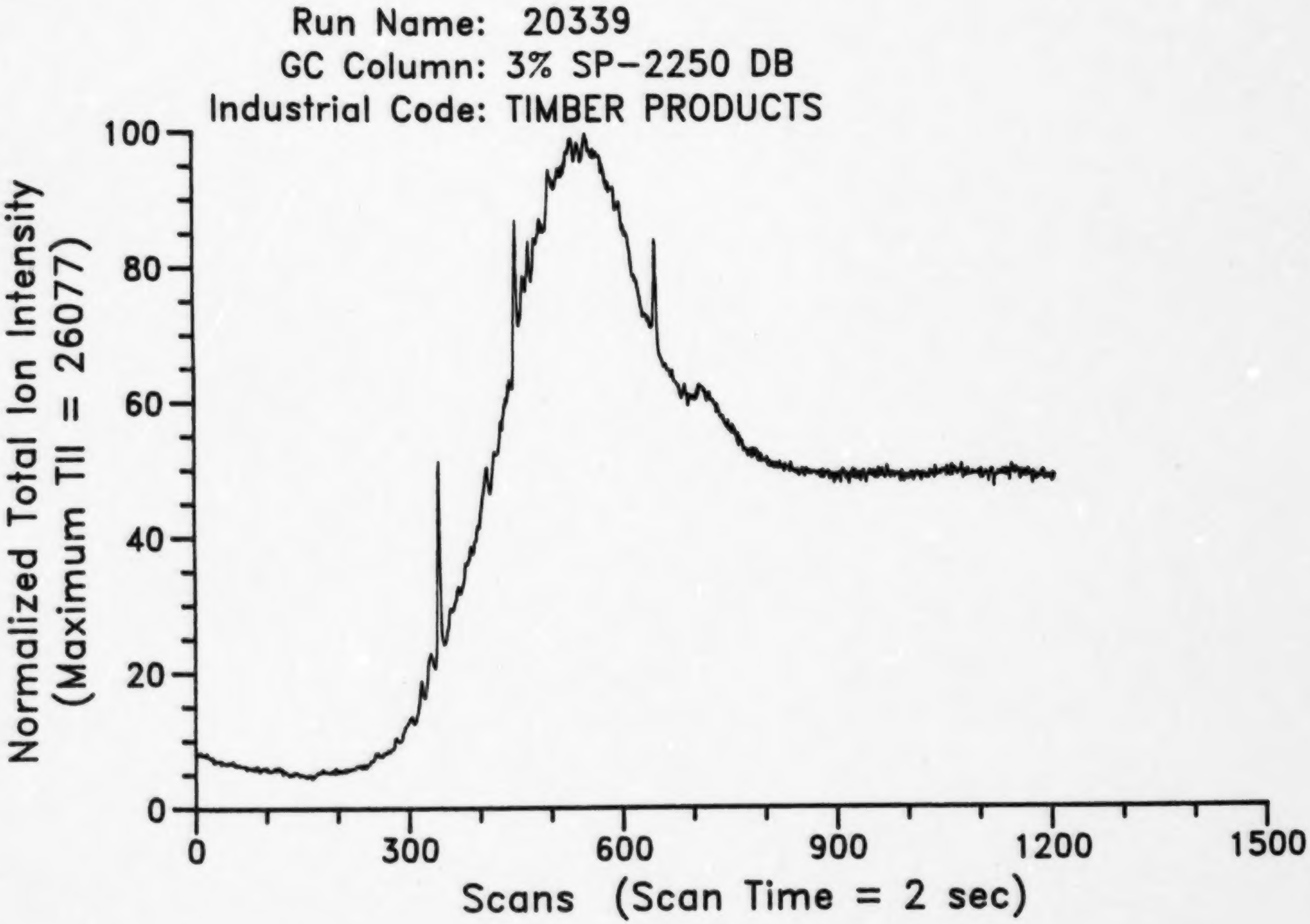


Figure B-165. Packed column chromatogram, extract 20339 (base/neutral), timber products industry.

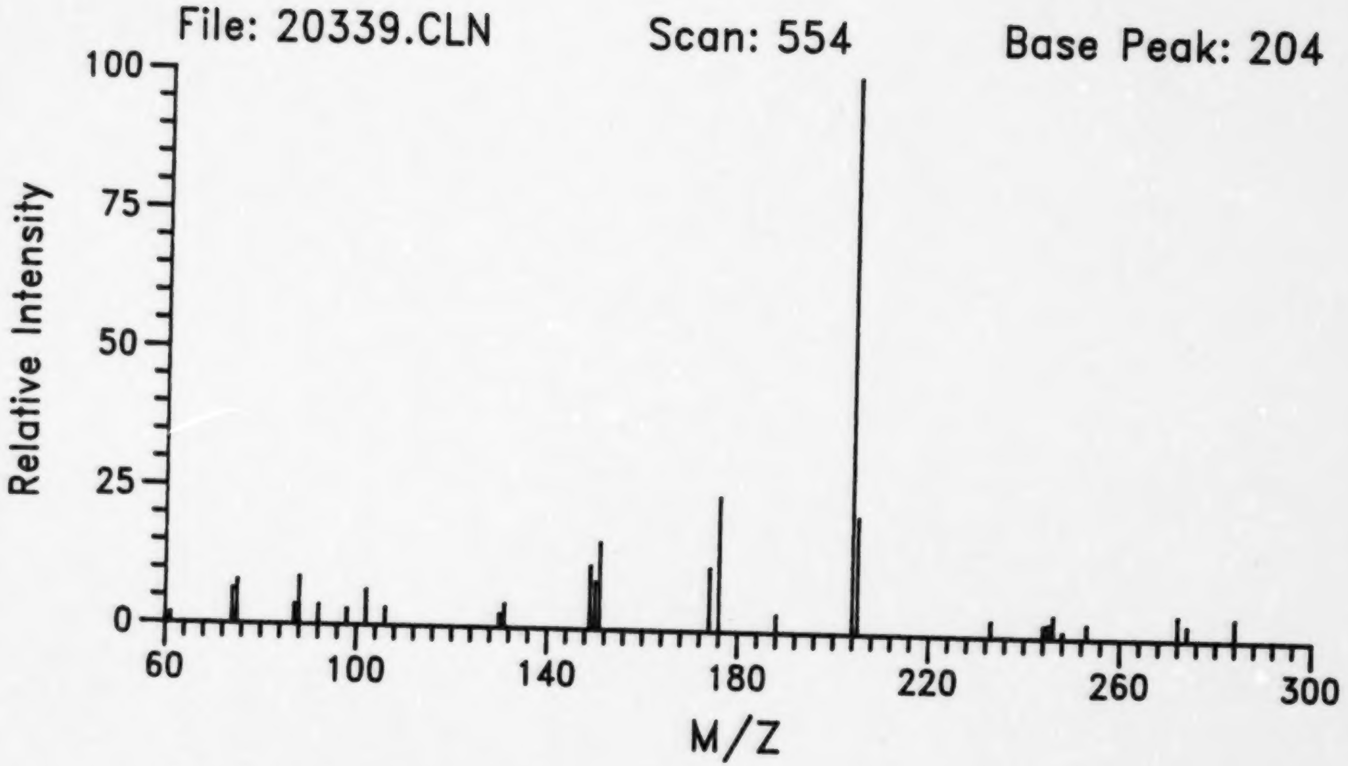


Figure B-166. Extract 20339 (base/neutral), timber products industry.
Frequency of occurrence: 9.

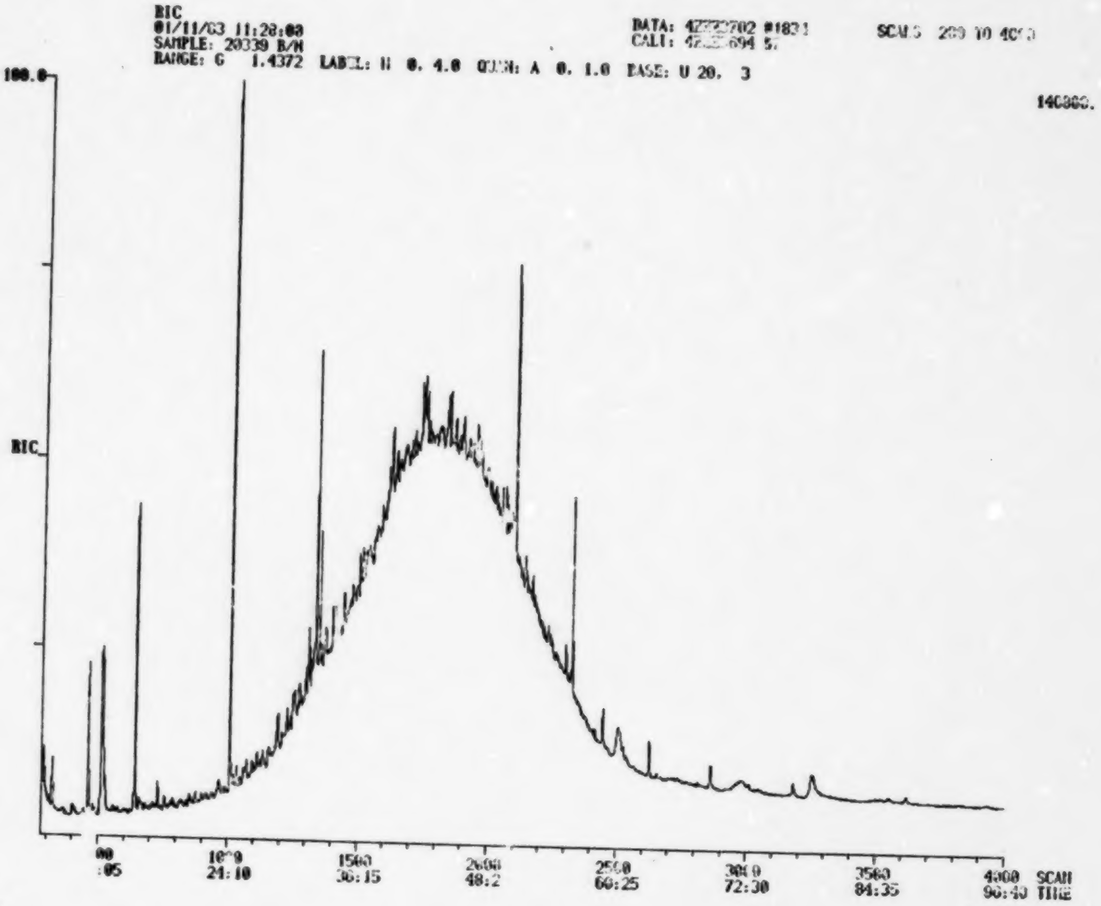


Figure B-167. Capillary chromatogram, extract 20339 (b/n), timber products industry.

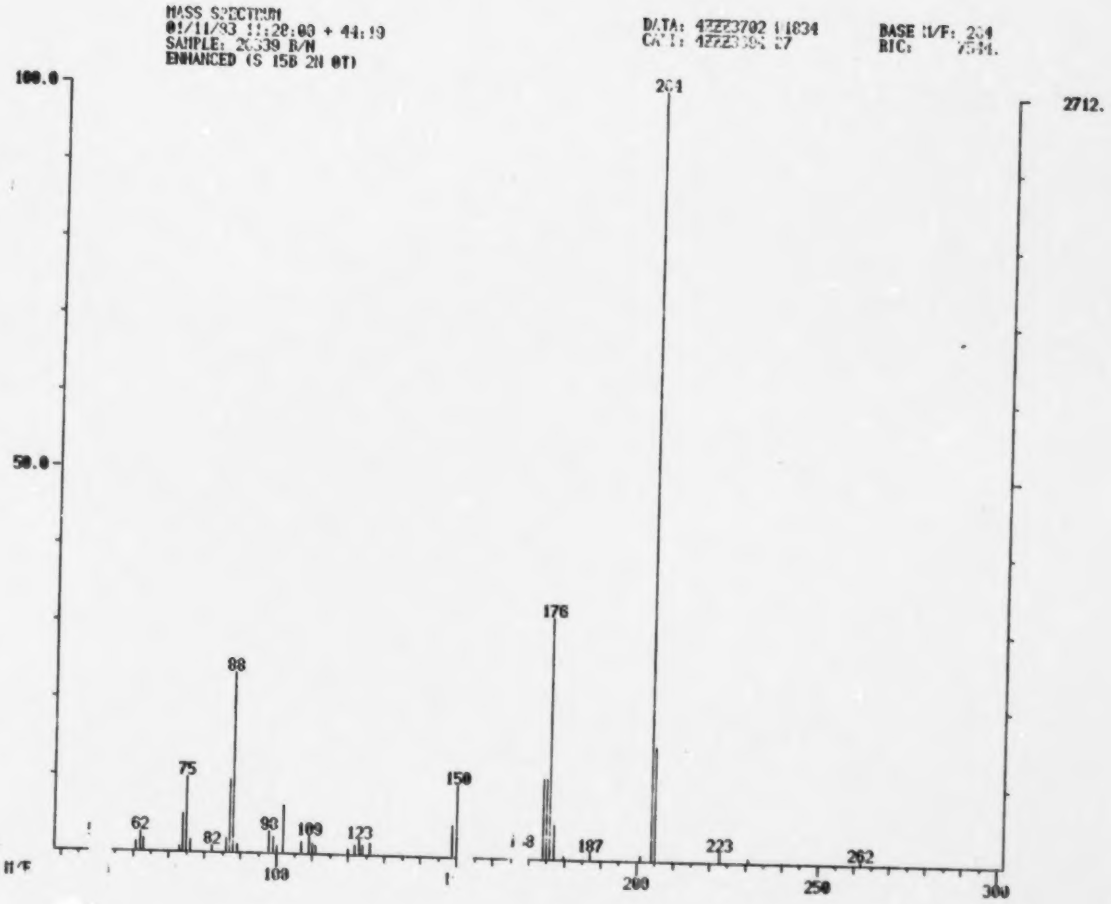


Figure B-168. Mass spectrum of EPA compound of interest, extract 20339, (base/neutral), timber products industry, scan #1834.

Figure B-169. Conclusions

From accurate mass measurement there are four possibilities: $C_8H_{12}O_6$ (error, -6.2 mmu); $C_7H_{12}N_2O_5$ (error, 5.0 mmu); $C_{14}H_8N_2$ (error, -0.9 mmu); and $C_9H_8N_4O_2$ (error, -4.9 mmu). Only the $C_8H_{12}O_6$ and $C_9H_8N_4O_2$ molecules can lose 28 mass units without requiring loss of N_2, a rare occurrence in mass spectrometry. Computerized library search presents a composition which includes nitrogen for four of the five top possibilities. There are no criteria for further selection. Functional group analysis would be essential for further information.

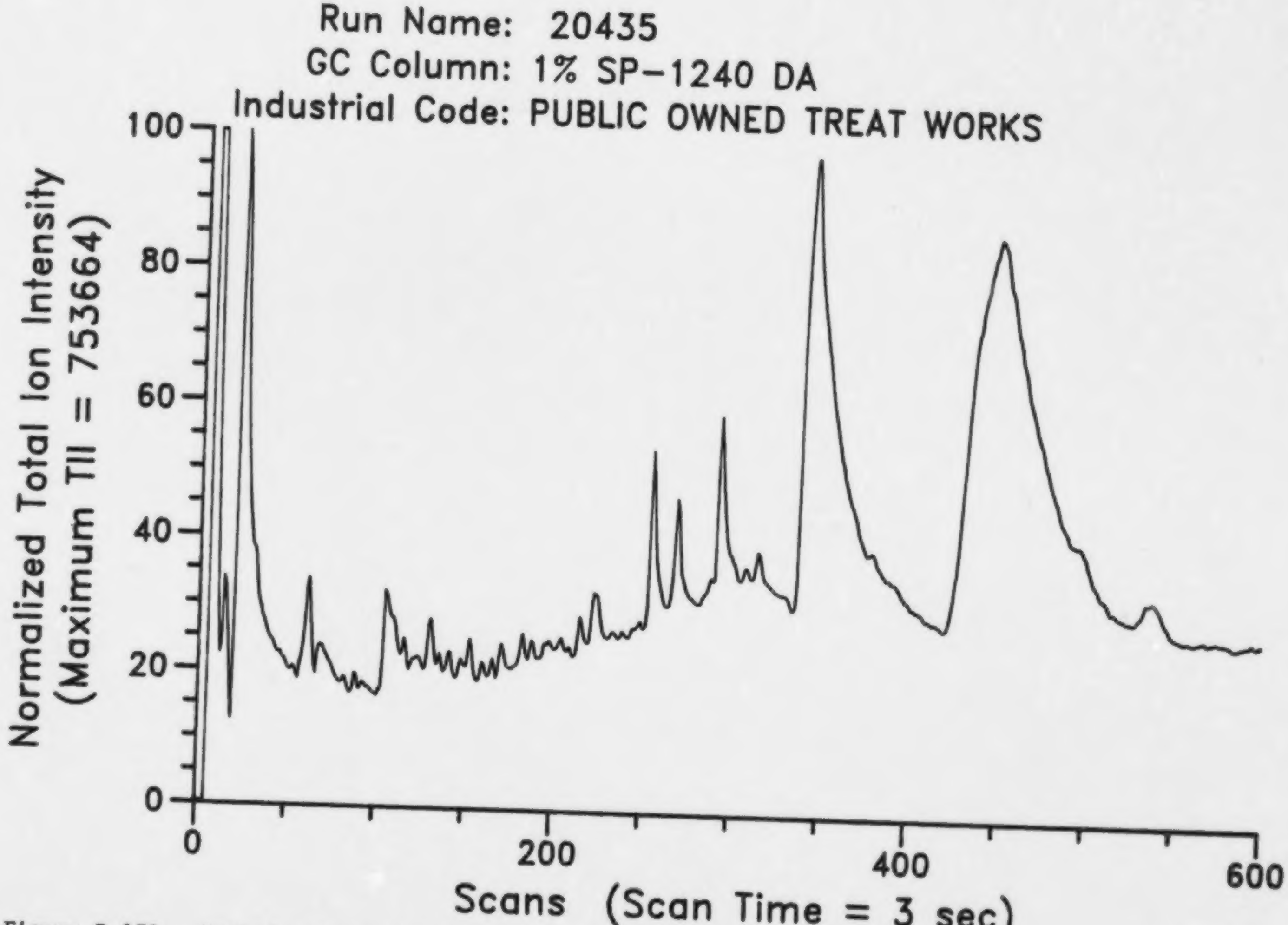


Figure B-170. Packed column chromatogram, extract 20435 (acid), publicly owned treatment works.

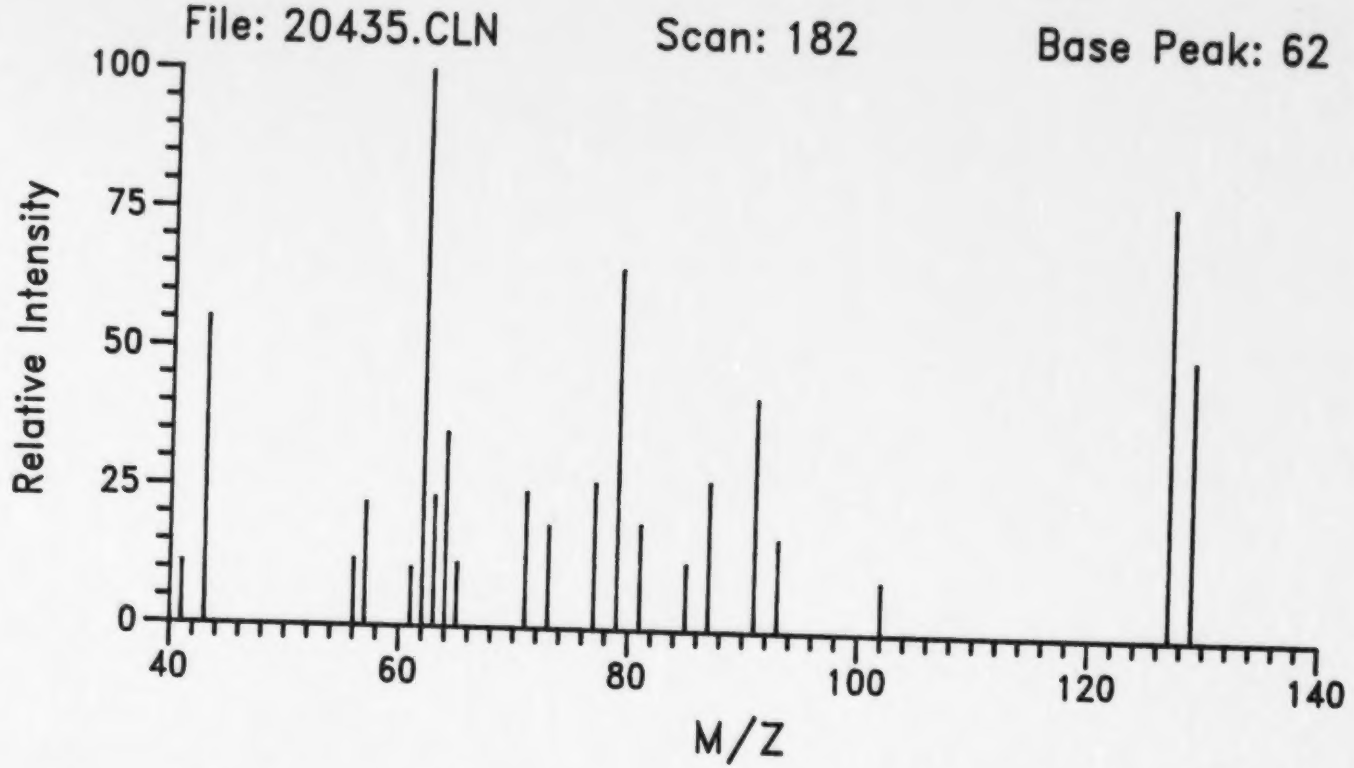


Figure B-171. Extract 20435 (acid), publicy owned treatment works. Frequency of occurrence: 33.

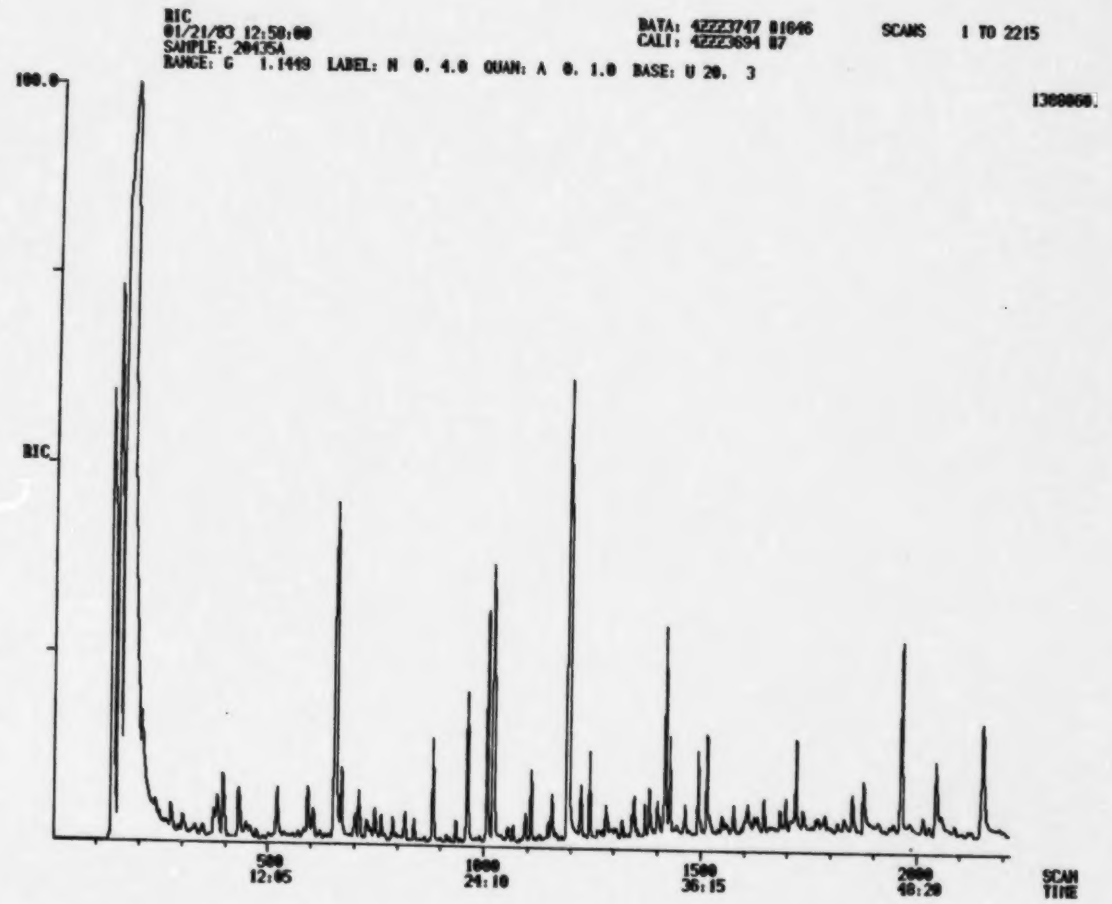


Figure B-172. Capillary chromatogram, extract 20435 (acid), publicy owned treatment works.

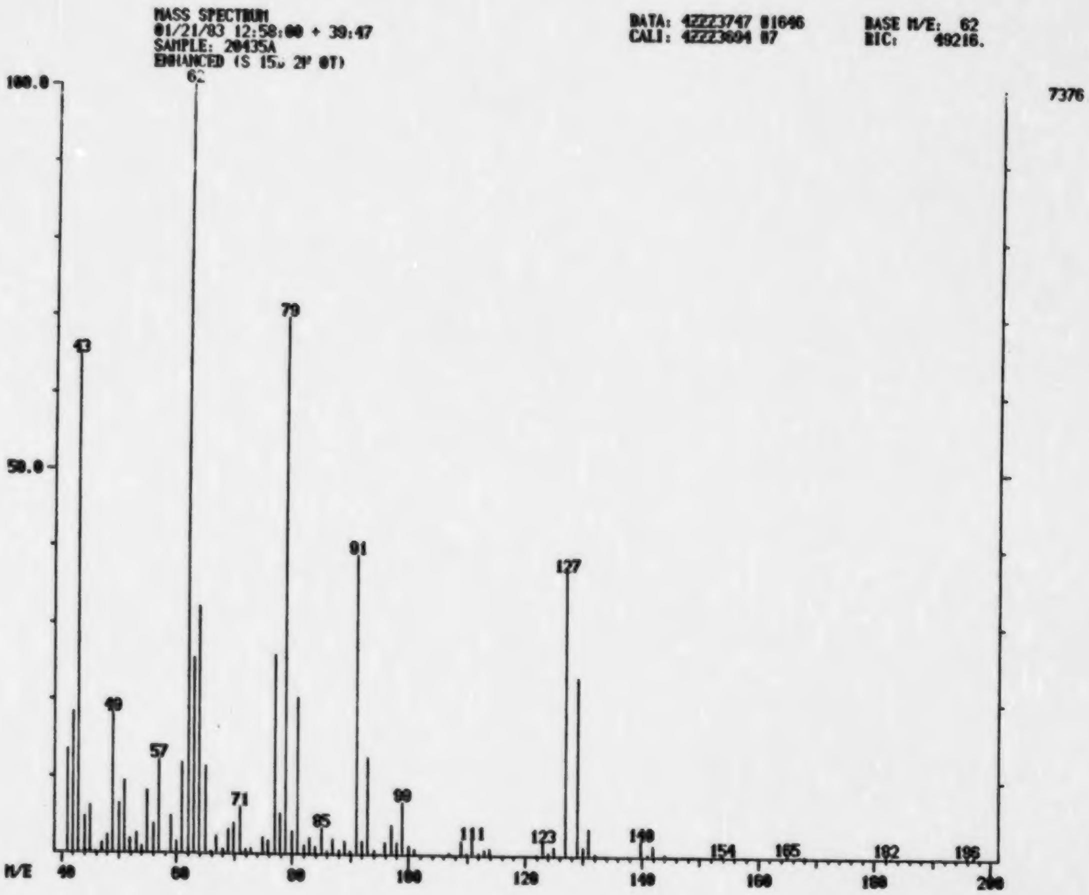
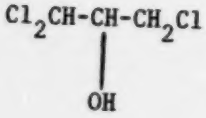


Figure B-173. Mass spectrum, EPA compound of interest, extract 20435 (acid), publicly owned treatment works, one isomer, scan #1646.

Figure B-174. Conclusions

Accurate mass measurement produces only one possibility for the ion of mass 127, $C_3H_5OCl_2$ (error, 0.1 mmu). Without nitrogen to account for the odd mass, this ion is not a molecular ion. Chemical ionization mass spectrometry was inconclusive. The cluster at mass 62 contains one chlorine atom and indicate a unit of $-CH-CH_2Cl$ in the molecule. The compound almost certainly contains at least one more chlorine atom and may contain more carbon as well. Perhaps a structure such as the one below is a possibility.



$$Cl_2CH-\underset{\displaystyle OH}{\underset{|}{C}H}-CH_2Cl$$

This compound would have a molecular weight of 162 and loss of H_2O would certainly be expected. Loss of water from 162 (producing mass 144) is not observed.

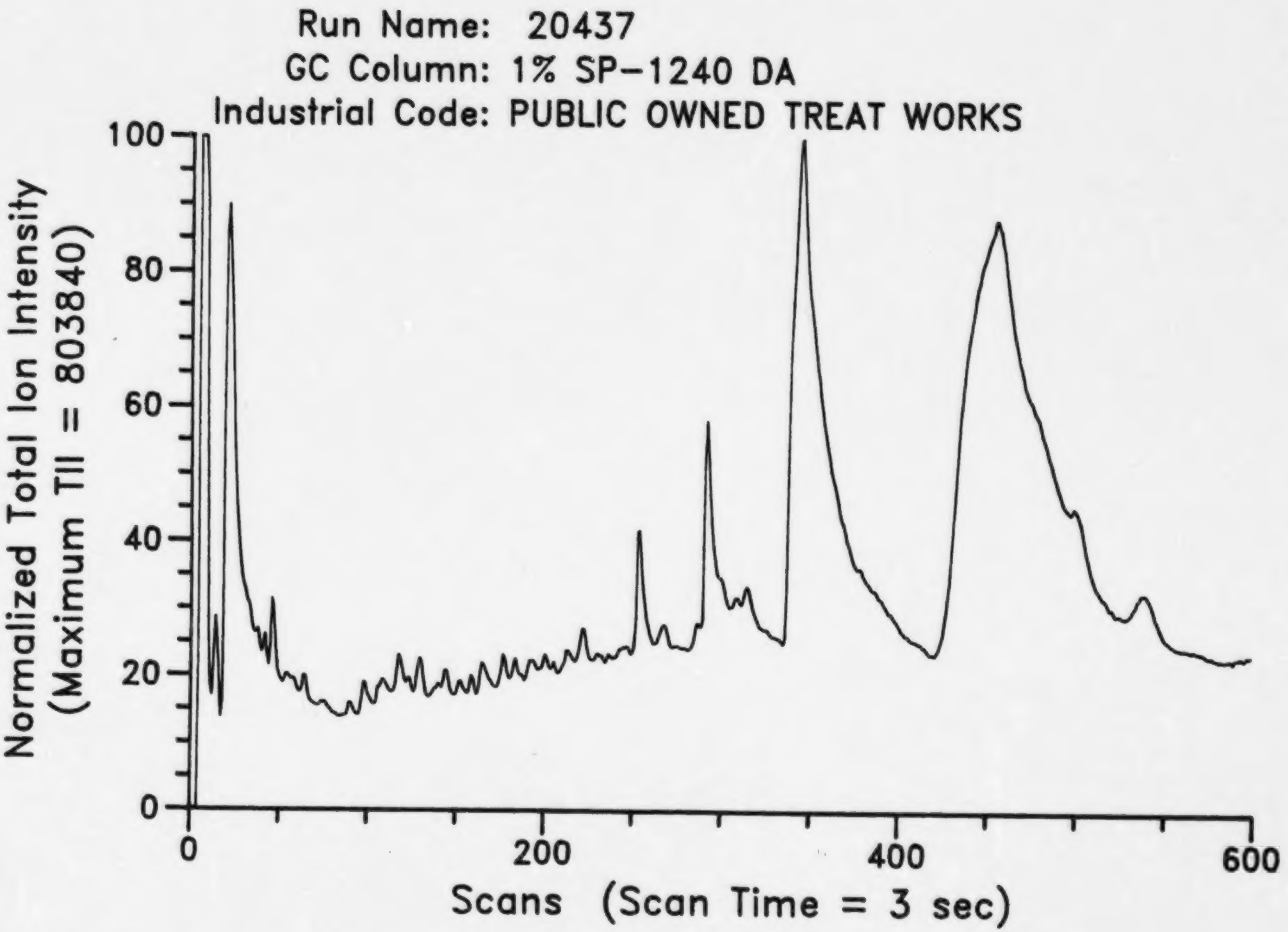


Figure B-175. Packed column chromatogram, extract 20437 (acid), publicly owned treatment works.

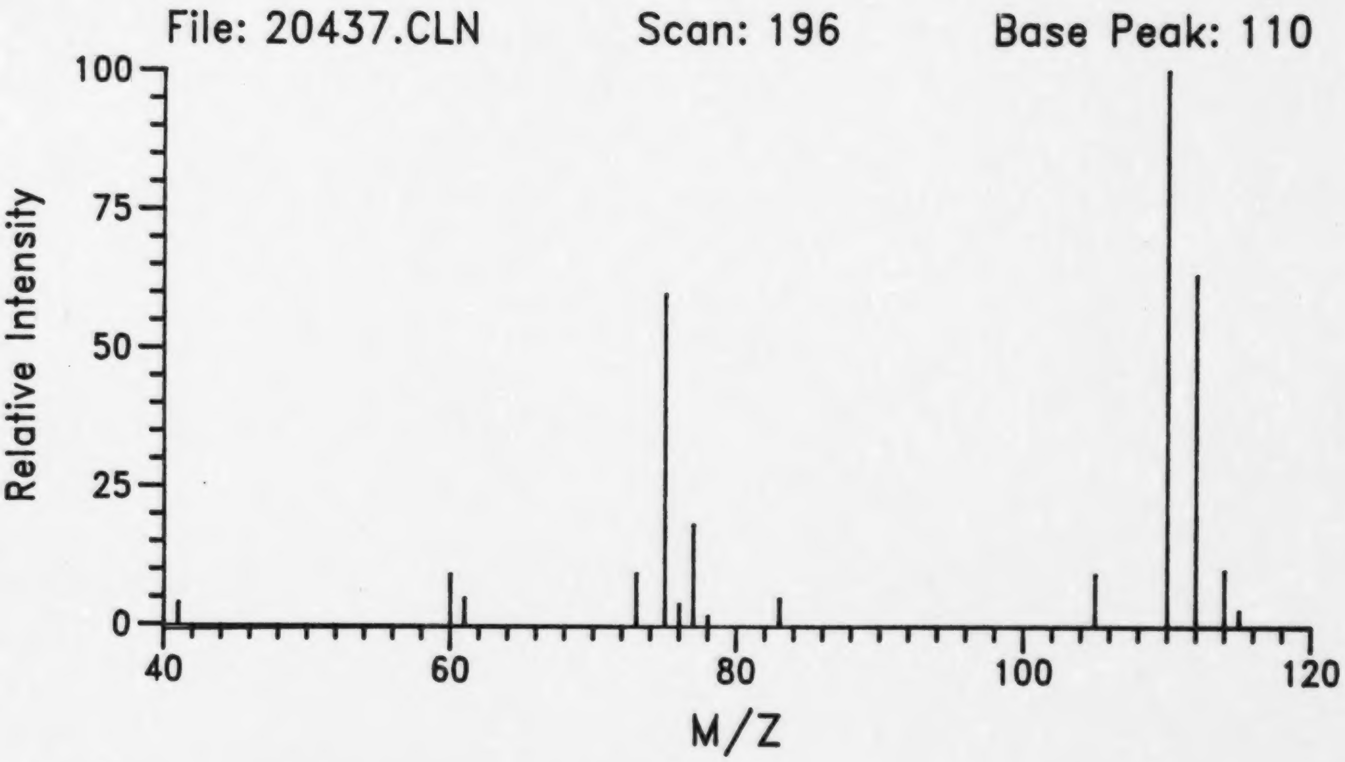


Figure B-176. Extract 20437 (acid), publicly owned treatment works.
Frequency of occurrence: 15.

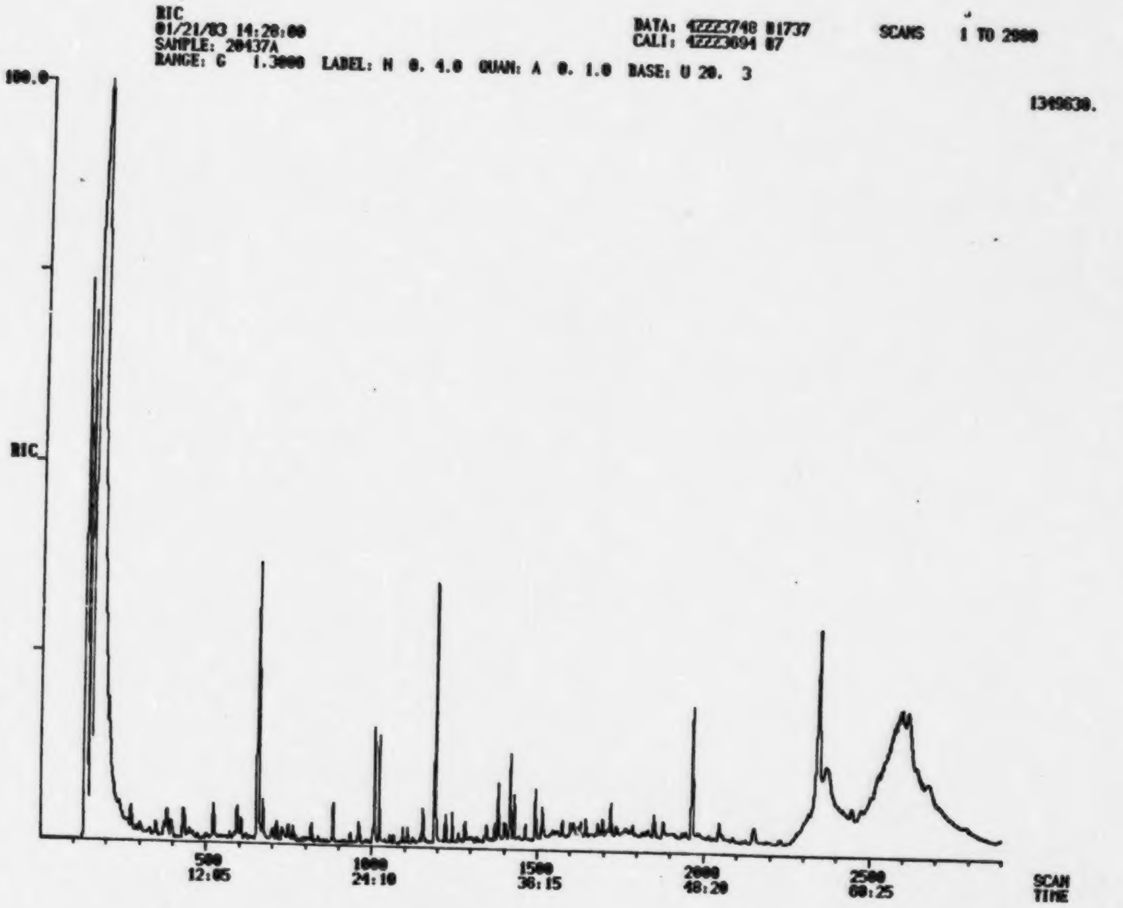


Figure B-177. Capillary chromatogram, extract 20437 (acid), publicly owned treatment works.

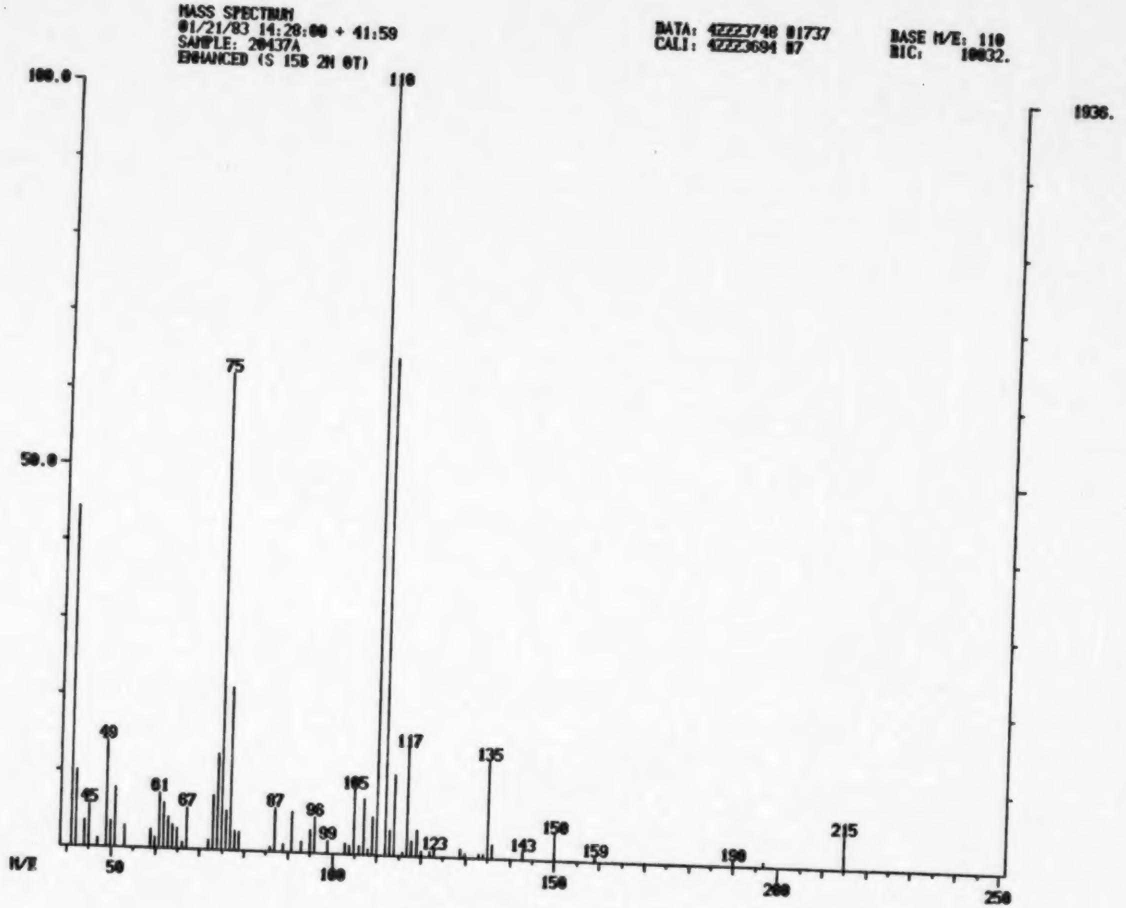


Figure B-178. Mass spectrum, EPA compound of interest, extract 20437 (acid), publicly owned treatment works, one isomer, scan #1737.

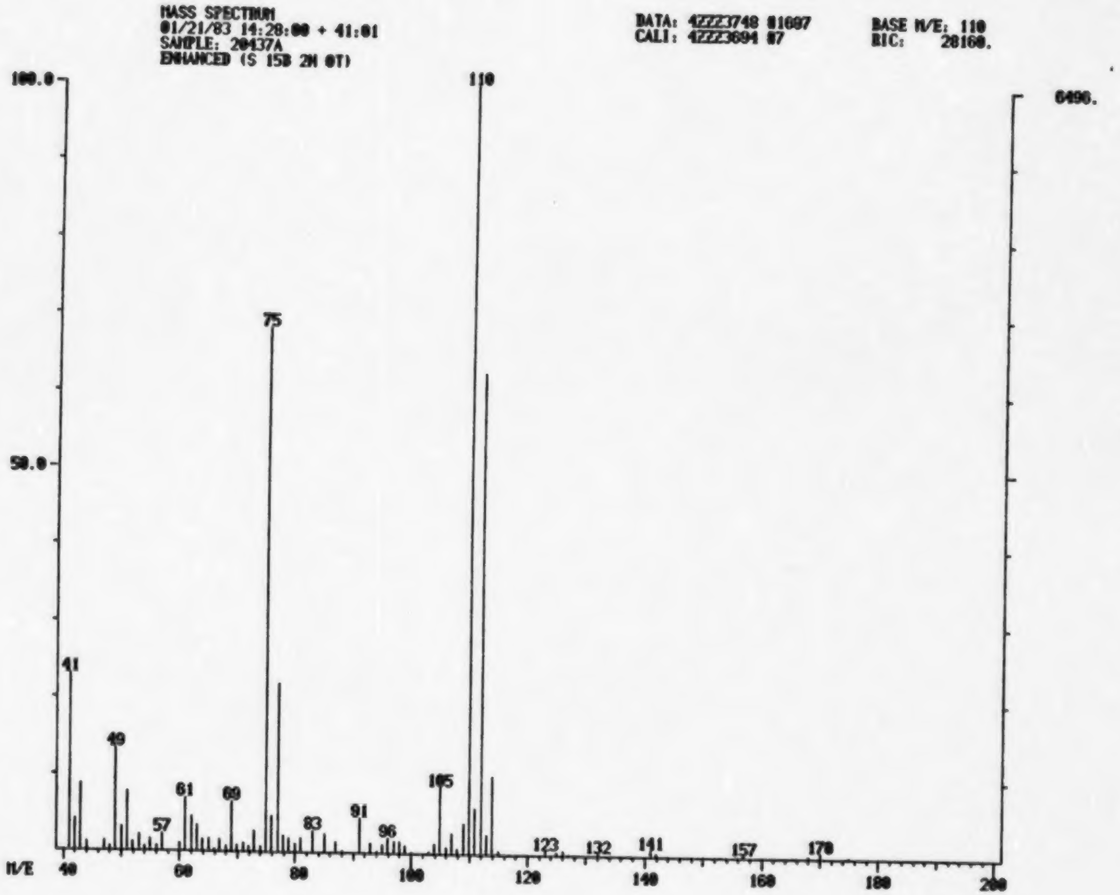


Figure B-179. Mass spectrum, EPA compound of interest, extract 20437 (acid), publicly owned treatment works, one isomer, scan #1697.

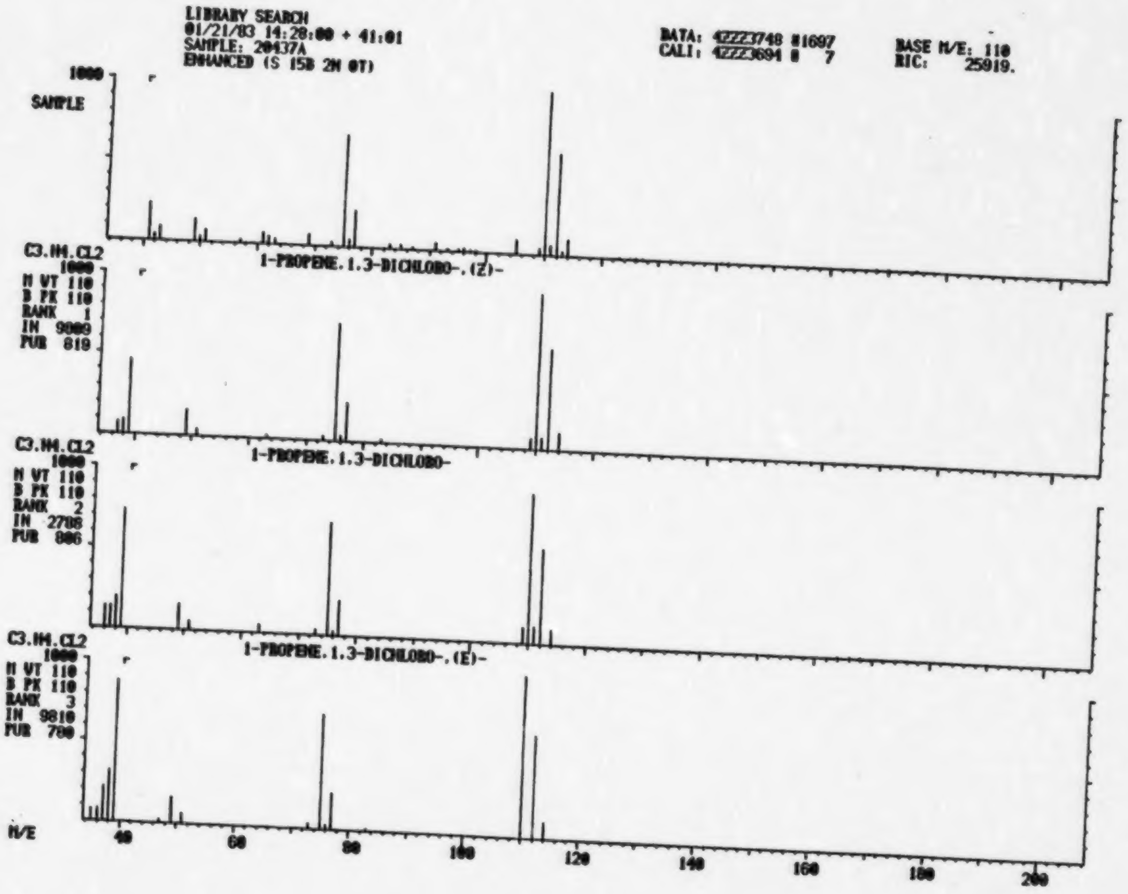


Figure B-180. Library search, EPA compound of interest, extract 20437 (acid), publicly owned treatment works.

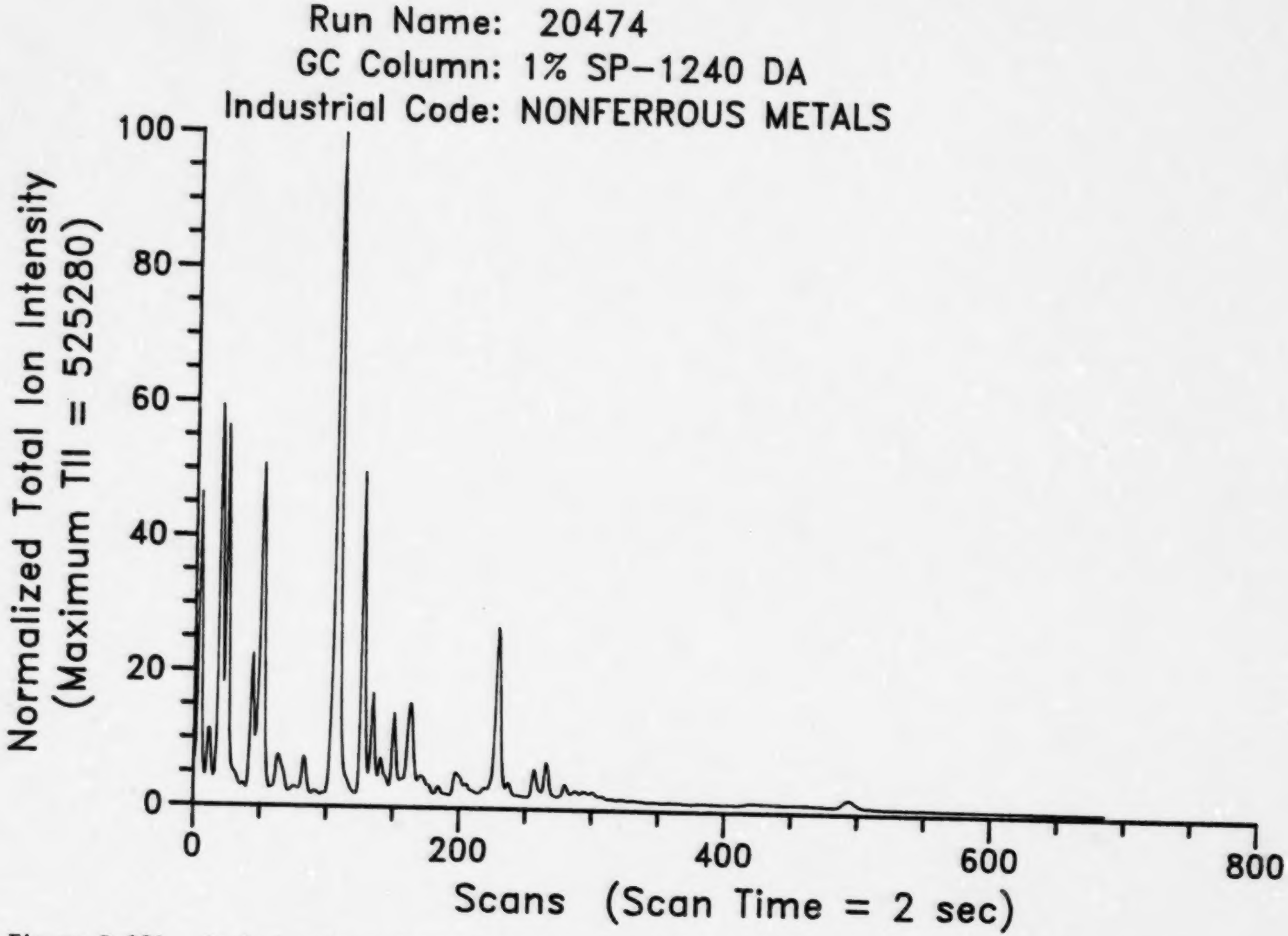


Figure B-181. Packed column chromatogram, extract 20474 (acid), nonferrous metals industry.

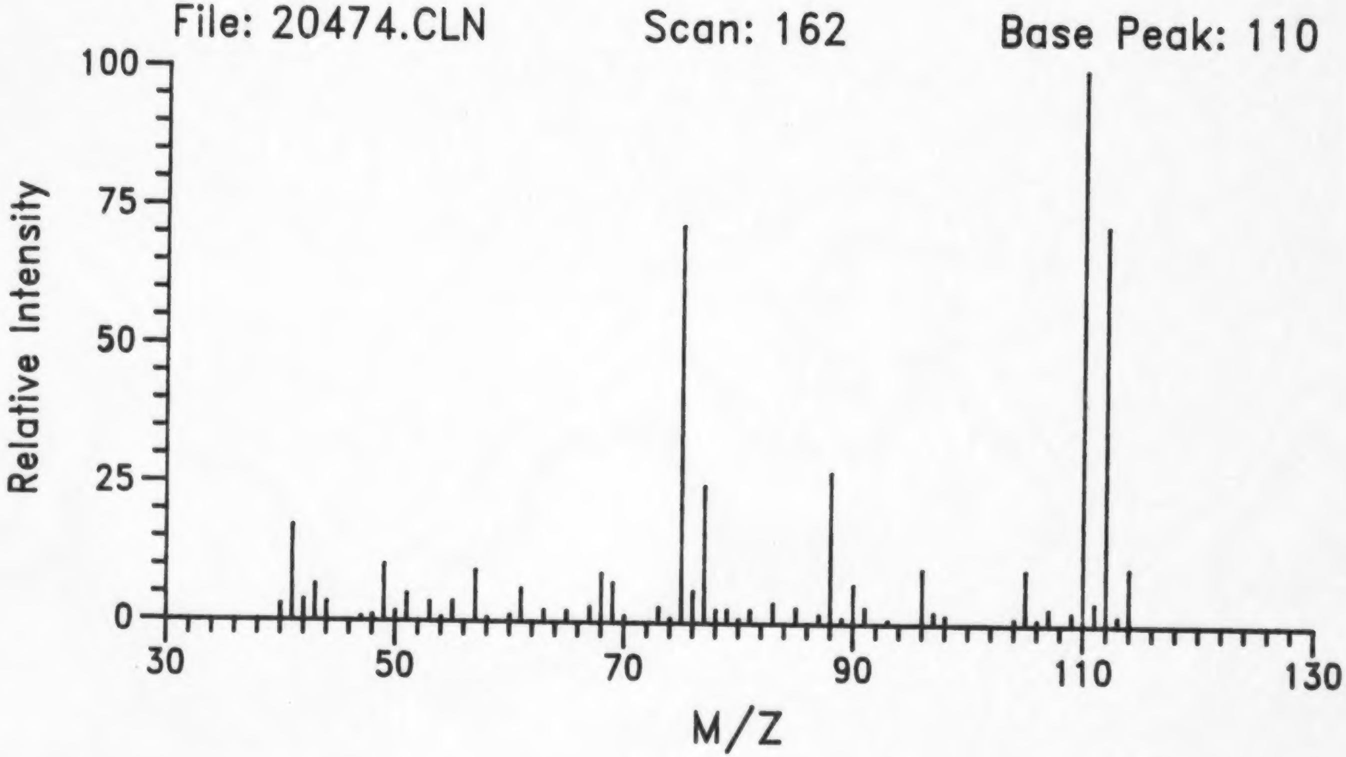


Figure B-182. Extract 20474 (acid), nonferrous metals industry. Frequency of occurrence: 8.

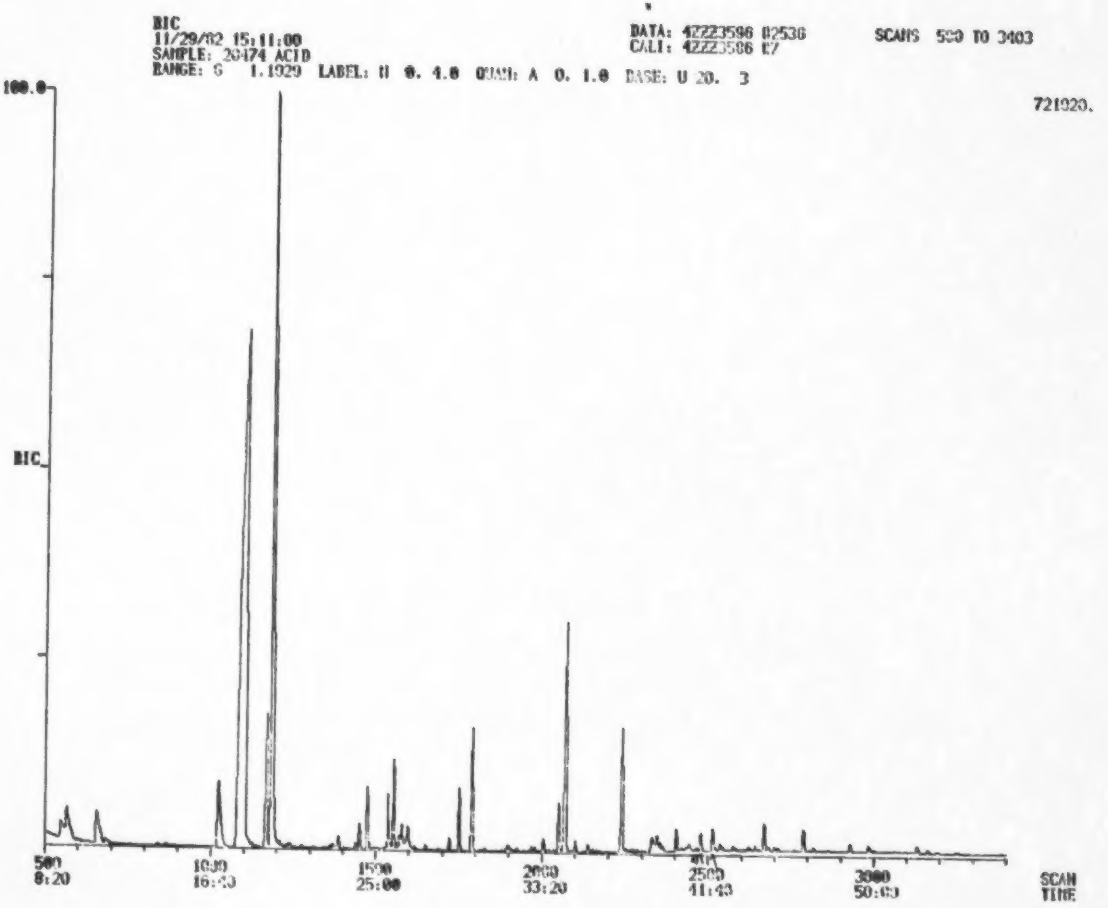


Figure B-183. Capillary chromatogram, extract 20474 (acid), nonferrous metals industry.

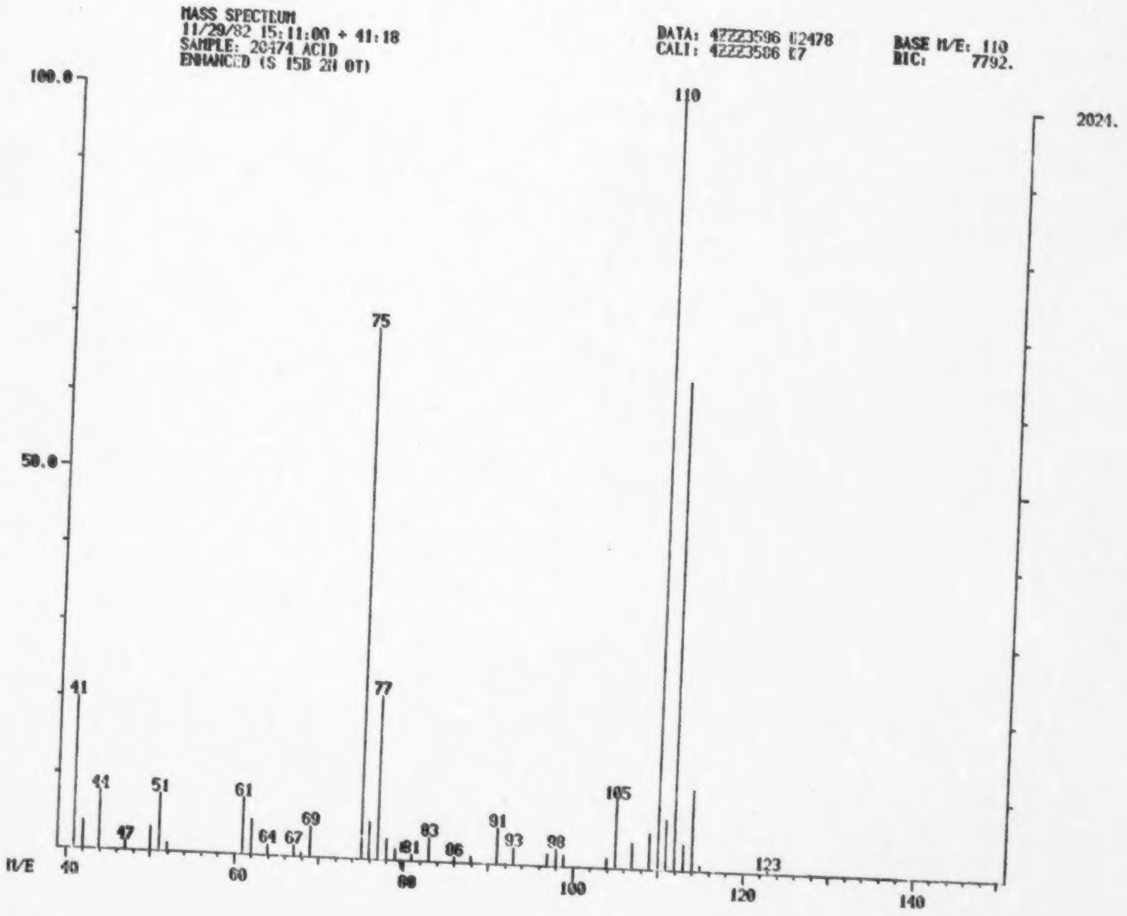


Figure B-184. Mass spectrum, EPA compound of interest, extract 20474 (acid), nonferrous metals industry, one isomer, scan #2478.

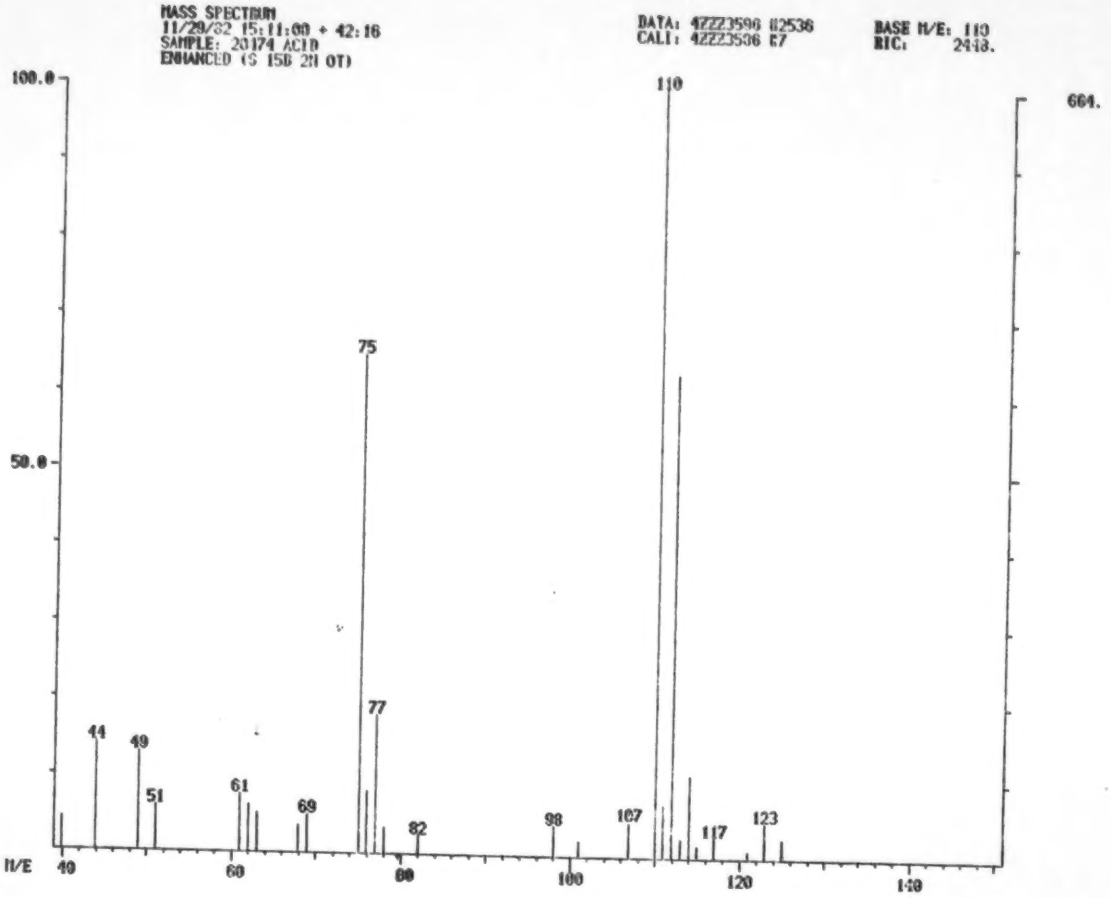


Figure B-185. Mass spectrum, EPA compound of interest, extract 20474 (acid), nonferrous metals industry, one isomer, scan #2536.

Figure B-186. Conclusions

Library search consistently (and with good FIT) identifies the compound of interest as 1,3-dichloropropene. However, this match does not account for the presence of an ion of mass 105 in the spectrum or account for a chromatographic retention time well beyond what would reasonably be expected for this compound. The molecular ion certainly contains more chlorine and may contain more carbon as well. The appearance of spectra in the two extracts, which correspond in every detail but differ widely in retention times, is a further confusing detail. The compounds of interest certainly contain two chlorine atoms and three carbon atoms, but, beyond these observations, no confident statements can be made.